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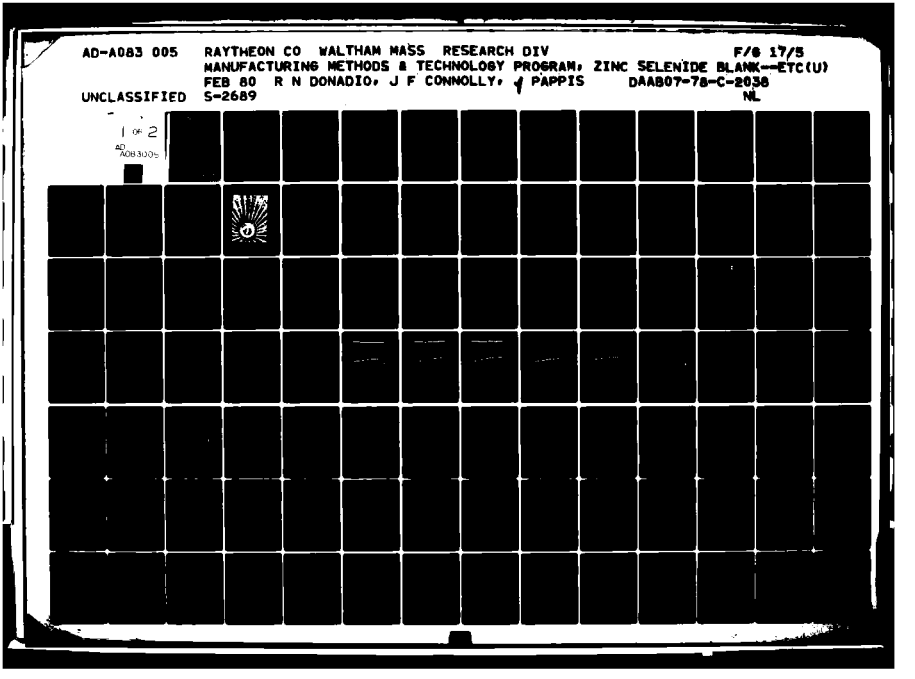

MANUFACTURING METHODS & TECHNOLOGY PROGRAM
ZINC SELENIDE BLANKS FOR WINDOWS AND LENS ELEMENTS

RAYTHEON COMPANY
RESEARCH DIVISION
WALTHAM, MA 02154

FEBRUARY 1980

FINAL TECHNICAL REPORT
17 July 1978 to 17 November 1979

Approved for public release; distribution unlimited.

Prepared for
US ARMY ELECTRONICS R&D COMMAND
NIGHT VISION AND ELECTRO-OPTICS LABORATORY
FORT BELVOIR, VA 22060

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This program established a fully automated process for the chemical vapor deposition of zinc selenide blanks. Prior to the program Raytheon Company had an established production process for the fabrication of this material. Further, Raytheon Company had, on an experimental basis, determined that the process could be fully automated by replacing the internally housed zinc source system with an externally housed system that fed zinc into the furnace at a predetermined rate. This system was successfully used in the Pre-Engineering, —>

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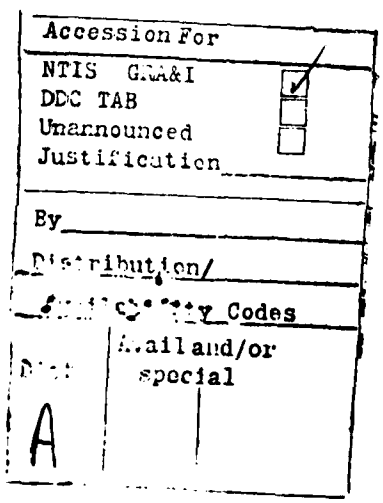
Engineering, Confirmatory, and Pilot deposits to yield zinc selenide with good optical and mechanical properties. The program also demonstrated that this process significantly reduced the costs of standard type lens blanks.



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GLOSSARY

Absorption Coefficient - Fraction of energy lost while traversing a pathlength of one centimeter through a material.

Chemical Vapor Deposition - A process by which chemicals are reacted in the vapor phase to form a compound.

Deposition Temperature - Temperature of the reaction zone in which the chemical vapor deposition takes place.

Evaporator - Apparatus used to form a vapor (or gas) from a solid (or liquid).

Flexural Strength - Maximum fiber stress a material will withstand before rupture in bending.

Image Spoiling Characteristics - That property of a transparent material that defines the ability to resolve discrete images.

Manifold - Apparatus used to distribute gas.

Molar Ratio - The ratio of the reactants entering the CVD process, based on molecular weight. In this program, the ratio of hydrogen selenide to zinc.

Retort - High temperature container used to hold liquid zinc.

Substrate - A form on which material is deposited, sometimes called a mandrel.

Torr - Millimeters of mercury - a measure of absolute pressure - 760 torr equal 1 atmosphere.

Feeder - Device for accurate control and injection of zinc into furnace.

Zinc Reservoir System - Apparatus containing one or several liquid zinc retorts.

1.0 INTRODUCTION

The purpose of this manufacturing and technology program was to establish a fully automated production process for the fabrication of high optical quality zinc selenide. The program also demonstrated production capability using this process and equipment.

Prior to the inception of the program the Research Division of Raytheon Company had successfully developed the techniques and facilities for producing state-of-the-art zinc selenide in large sizes using the chemical vapor deposition (CVD) process. This program, sponsored by the United States Army Electronics Research and Development Command, investigated further automation of an existing process with the aim of reducing the cost of a standard lens blank, and showing the production capability at the required rate. To achieve the type of automation desired Raytheon Company used techniques that they had previously shown, on a non-production basis, to be feasible.

The program spanned seventeen months and consisted of two phases. In the first phase, the zinc reservoir system was replaced with an automated external zinc supply. This system was used in the pre-engineering, engineering, and confirmatory deposits and the optical and mechanical properties of zinc selenide produced were evaluated. The concept of depositing zinc selenide on a curved substrate was introduced in the confirmatory deposit as another method of reducing the cost of a lens blank. The second phase of the program demonstrated the production capability of a pilot line to manufacture high quality zinc selenide blanks at the rate of four-hundred and eight-one (481) units per month.

2.0 NARATIVE AND DATA

2.1 CVD Zinc Selenide State-of-the-Art System

The chemical vapor deposition (CVD) process can be understood as follows: volatile compounds or other volatile species of the material to be deposited are passed into the deposition zone of a furnace that has been heated to some predetermined temperature and is held at some predetermined pressure. The volatile compounds (or elemental vapors) are directed to the substrate (mandrel) and are then allowed to thermally decompose and react at the substrate to form the solid material. Typical deposition rates are 0.005 to 0.010 in./hr. The reaction products are pumped away and disposed of through an exhaust system which entraps particulate matter from the furnace exhaust and neutralizes any unreacted gases. A monitor system ensures that no pollutants are released into the environment.

In the state-of-the-art system for the CVD of zinc selenide, the zinc source is contained in graphite reservoirs which are located at the bottom of the furnace. Zinc vapor is generated by heating the zinc to a liquid and then evaporating it at a reduced pressure. The vapors can be carried into the deposition zone by using a carrier gas. Hydrogen selenide gas is passed directly into the reaction zone through appropriate gas inlets. A chemical reaction occurs inside the substrate box, resulting in a crystalline deposit of zinc selenide on the sides of the substrate. Figure 1 displays a flow diagram for this process.

The reaction used in depositing zinc selenide is:

$$Zn(v) + H_2Se(v) \rightarrow ZnSe(s) + H_2(v)$$

$$\Delta G_f^{\circ},\ 750°C = -36.7 \text{ Kcal/mole}$$

$$\Delta G_{RX},\ 750°C = -23.2 \text{ Kcal/mole}$$

The value of ΔG_{RX} is the energy for the reaction at a specific deposition

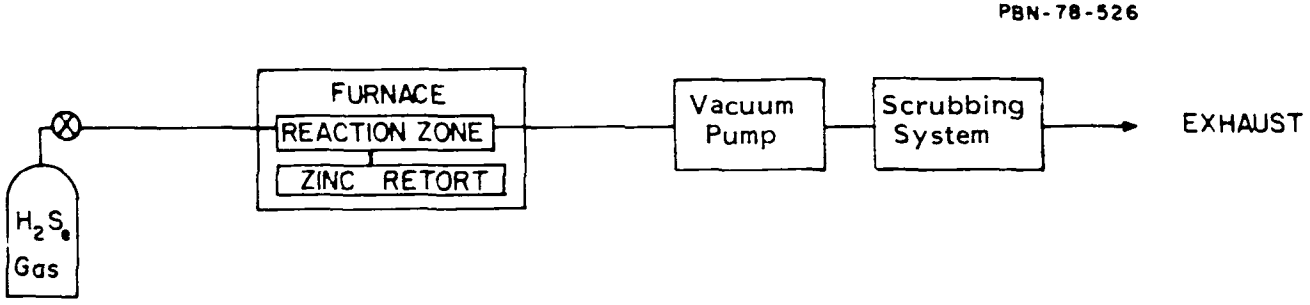

Figure 1. Flow Diagram for Reservoir System.

temperature (750°C), pressure (40 torr), and reactant molar ratio (H_2Se/Zn = 1.1).

A schematic representation of the state-of-the-art system is presented in Figure 2, while Figure 3 displays the 48-inch access diameter of the furnace used in the program. This furnace system is capable of producing zinc selenide in thicknesses of one inch over approximately 6,000 square inches, although smaller area deposits are more common. Since deposits of the thickness required in this program can be made in less than two weeks, it is possible to achieve a production rate approaching 12,000 square inches per month, if necessary.

The system described has been automated to a large degree. For example, temperature, furnace pressure, and all gas flows use automated controls. The one sub-assembly in the larger furnace that has not been fully automated is the zinc reservoir system. Automation of this sub-system has been difficult due to the large volume of zinc (~one-half ton loads) required for some deposits. There are advantages and disadvantages to the existing system. For example, if the operating conditions of the system are close to the desired range,most cyclic changes that occur as a function of time, result in only minimal material variations because of the long time needed to cause changes in the large mass of zinc being monitored. On the other hand, if one desires to alter the operating conditions the long lag time encountered can be a disadvantage since over two hours are required to sense the effect of the changes made. Further, small changes in retort conditions (pressure, temperature) can result in significant changes in the zinc usage rate. On balance, therefore, there are obvious advantageous to automating this part of the system.

2.2 External Zinc Source System

Prior to this program Raytheon Company had experimented on a simple, more accurate method of supplying zinc vapor to the reaction zone of the furnace. The apparatus was designed, built, and used on non-production

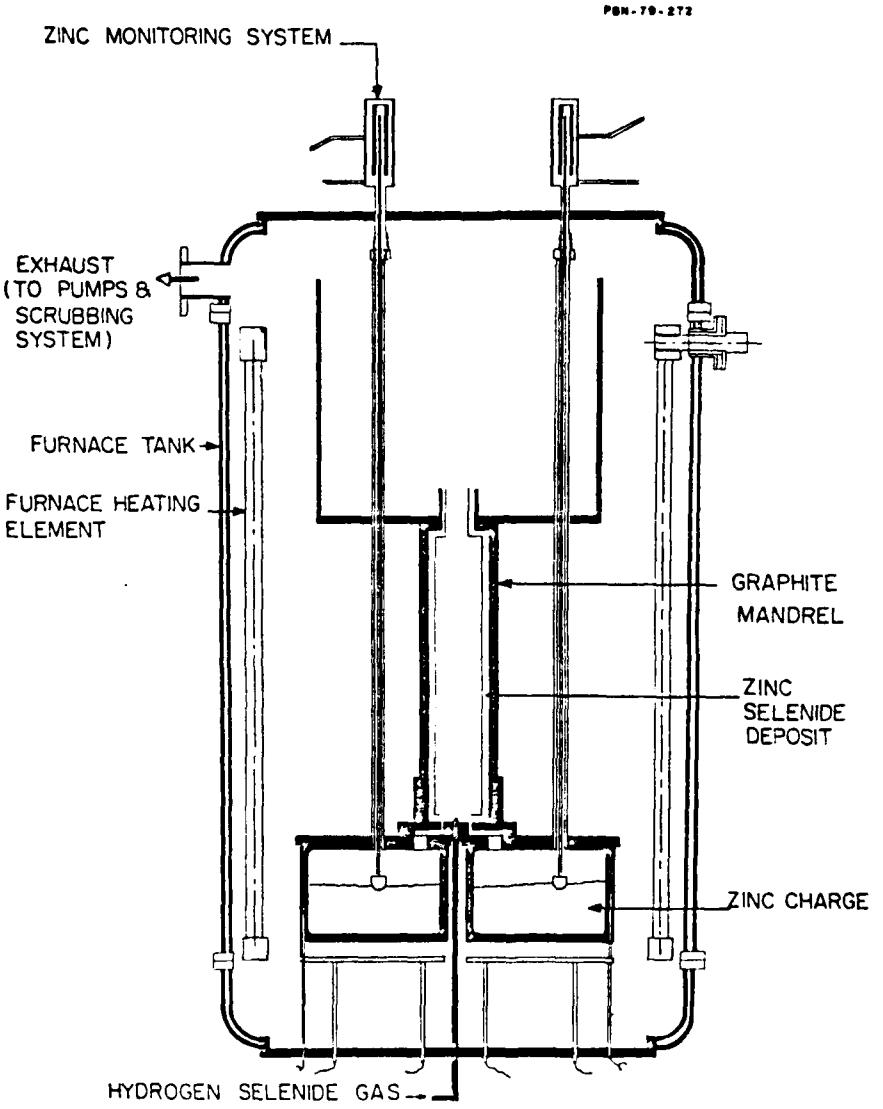

Figure 2. **Schematic Diagram of CVD ZnSe Furnace.**

Fig. 3 High Production Rate Vacuum Furnace.

type runs prior to the beginning of this program. It was adapted for use on this program. In the technique developed, zinc is introduced into the furnace in wire form, vaporized, and introduced into the reaction chamber. The feeder can deliver over 2,000 g/hr of zinc, which is more zinc than has been used in any zinc selenide deposit to date. A flow diagram for the wire feeder assembly is presented in Figure 4.

A major advantage of this system is the automatic control of accurate volumes of zinc. With the use of mass flowmeters for the hydrogen selenide gas, and the feeder system for the zinc, the zinc selenide reaction can be controlled accurately to any desired molar ratio.

The elimination of bulky retorts also makes available additional space for larger mandrels or substrates.

2.3 Experimental Deposits

2.3.1 Introduction

Original scheduling of the program called for the use of the state-of-the-art system in the engineering deposit, while a large size feeder system was being fabricated. The feeder was then to be incorporated in the confirmatory deposit after testing and debugging. However, the feeder system was fabricated by Raytheon and available at the start of the program. Several experimental deposits were conducted to evaluate the performance of this system.

2.3.2 Depositions

In the first experimental deposit it was observed that as a result of

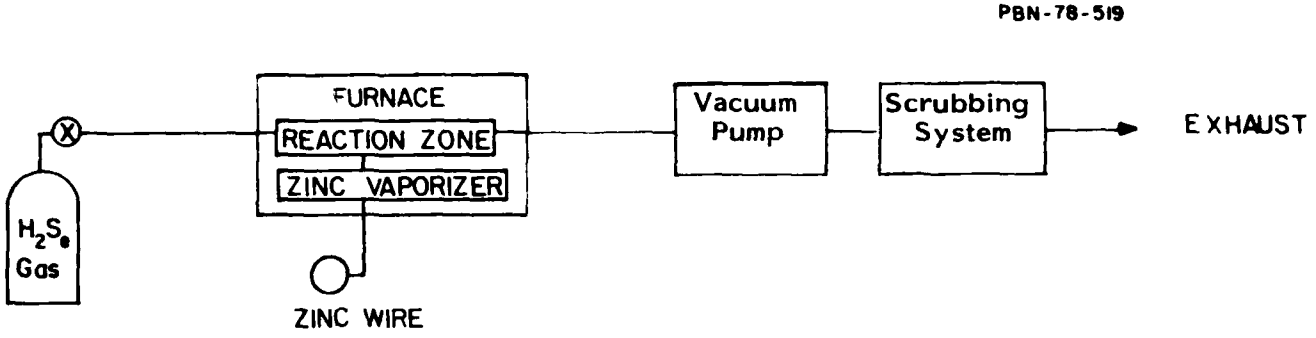

Figure 4 Flow Diagram for Wire Feed Assembly.

a broken component in the evaporator system, the zinc melted in the evaporator and flowed into the furnace rather than being transferred into the reaction chamber. The broken component apparently failed as a result of being rigidly restrained with no freedom for thermal expansion. An expansion joint was added in the feeder chain inside the furnace resolving this problem in subsequent deposits. No material was deposited in this first run.

In the second experiment, a condensation problem was observed in the outlet section of the evaporator. The temperature in this area was low enough to cause the zinc vapor to condense resulting in a blockage in the evaporator. Additional thermal insulation was added to this section for the following deposit and proved to be quite successful. More thermocouples were also placed in critical areas to ensure that the temperature could be accurately monitored.

The zinc feeder system performed successfully in the third experiment, a 67 hour deposit. The desired vaporization rates were easily maintained throughout the run. Examination of the evaporator system after deposition showed no evidence of zinc condensate or system malfunctioning. The resultant deposit was 150-200 mils deposited uniformly over the entire mandrel.

2.4 Pre-Engineering Deposit

An 84 hour zinc selenide deposit was conducted prior to the engineering sample deposit to further evaluate the performance of the deposition system. It was also felt that a moderately long run should be made to yield zinc selenide with sufficient thickness to measure the optical and mechanical properties of material made by this method. The deposit was made in a 17 by 9 by 60 inch graphite box mandrel. The deposition conditions for this pre-engineering deposit are presented in Table 1.

The resultant deposit showed adequate overall thickness and thickness distribution. Figure 5 displays a thickness profile for Plate A, one of the two

TABLE 1

DEPOSITION CONDITIONS

Run No.	Temp (°C)	Furnace Pressure (torr)	H_2Se Usage (lpm)	Zinc Usage (g/hr)	Deposition Time (hrs)
Pre-engineering	730	24	4.0	700	84
Engineering Deposit	730	24	4.2	735	327
Confirmatory Deposit	750	25	5.0	874	143
Pilot Deposit	750	24	5.25	917	208

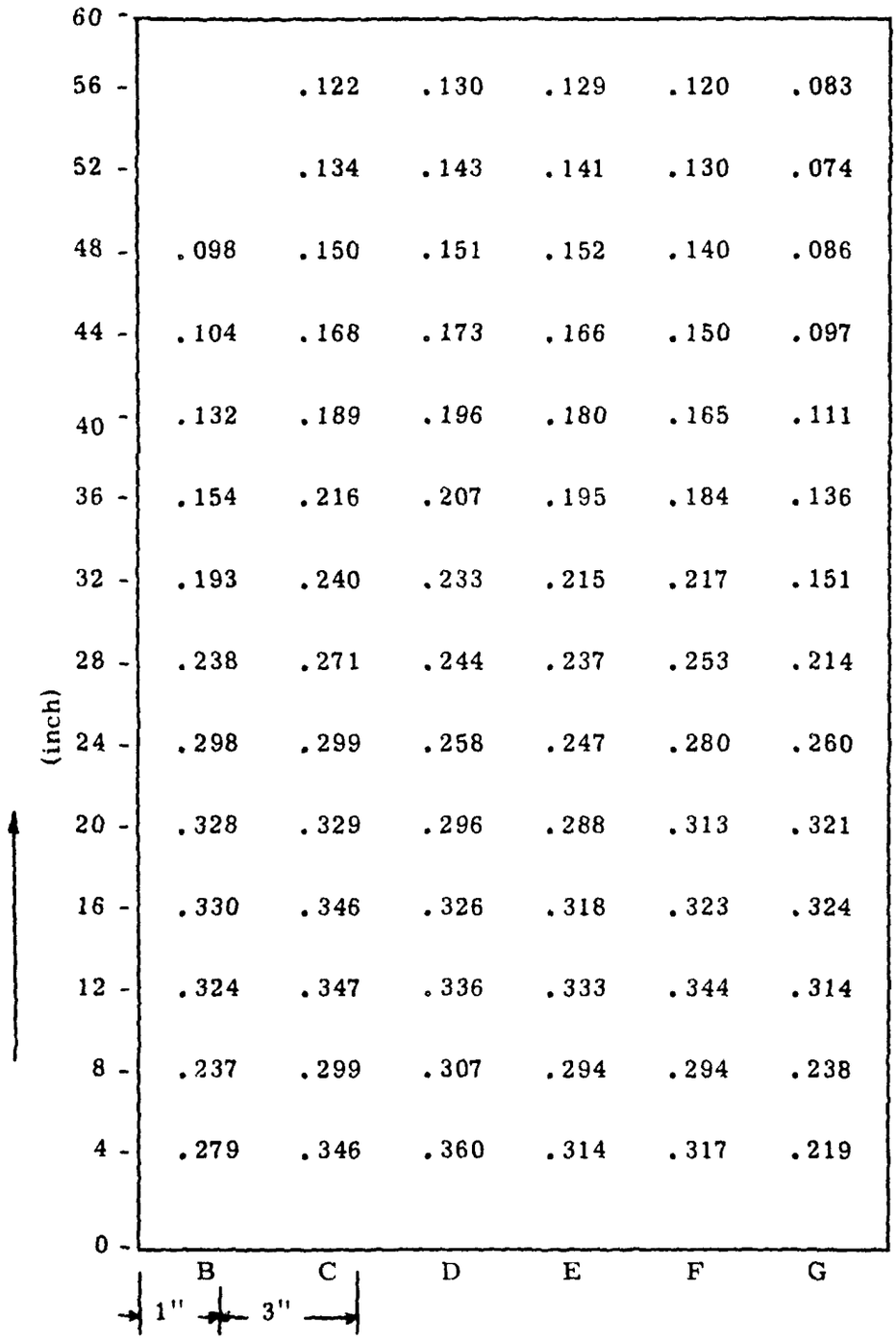

Figure 5. Thickness Profile, Plate A, Pre-Engineering Deposit.

17 by 60 inch plates deposited. Test samples were taken from the top, middle and bottom of the deposited plates and tested for infrared transmittance. Figure 6 presents the infrared transmittance curve for one of these three locations, all of which were equivalent. The average absorption coefficient at 10.6 micrometers (including surface absorption) was measured to be $\sim 0.001\ cm^{-1}$.

Flexural test specimens were prepared from the top and bottom sections of the deposit. The zinc selenide was fabricated into 1/8 by 1/4 by 2-1/2 in. beams and tested in flexure using 4-point loading over a 2 in. span. The results presented in Table 2 show slightly higher strength values than those typically observed. The 730°C deposition temperature for this deposit is the main reason for the increase in strength. A finer grained structure is produced at this lower deposition temperature, yielding a higher fracture strength. (Standard deposition temperature zinc selenide is 750°C - producing ~7500 psi fracture strength).

Examination of the evaporation system after the deposit showed no evidence of leakage to the surrounding furnace, nor zinc condensate in the evaporator chain. No malfunctions of any kind were detected throughout the deposit.

2.5 Engineering Sample Deposit

2.5.1 Sample requirements

From this deposit the program required that 12 chemical vapor deposited zinc selenide blanks be fabricated and tested. The blanks fabricated were 75 mm in diameter by 9.7 mm thick. Dimensional tolerances on all samples were $^{+2}_{-0}$ mm. The blanks were tested to determine if they met the following specifications: (Description of test equipment and test procedures are presented in the Appendix.)

a) Transmittance - The uncoated transmittance of the blanks shall be greater than 58% over the wavelength region 8 to 13 micrometers at normal

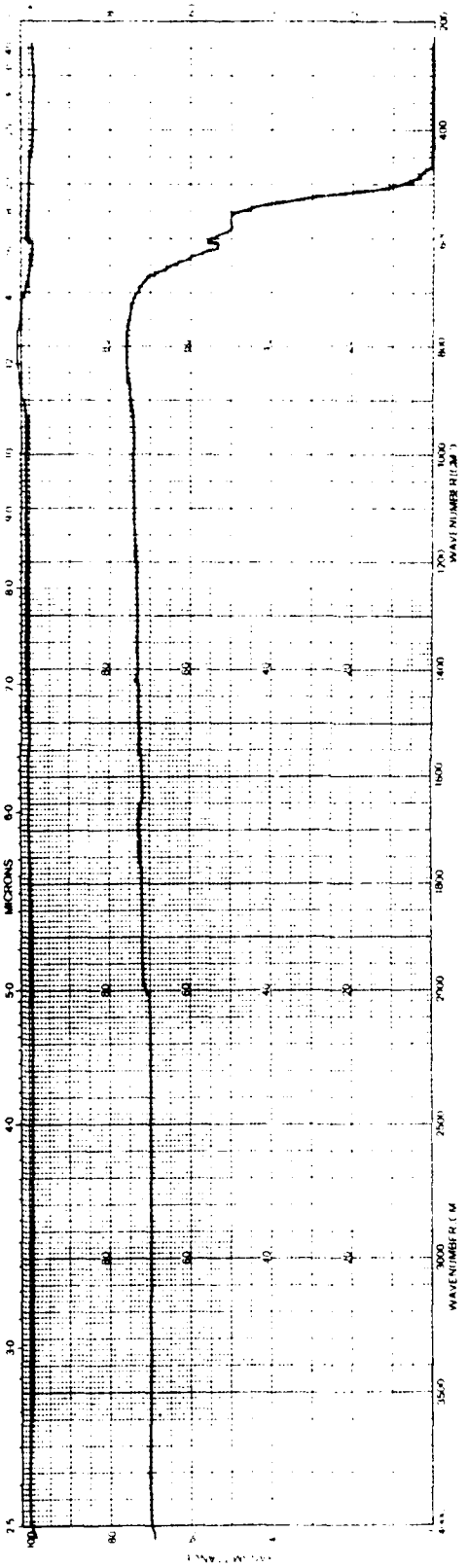

Figure 6. Infrared Transmittance, Plate C, Position 2M, t = 0.261 in., Pre-Engineering Deposit.

TABLE 2

FLEXURAL STRENGTH
PRE-ENGINEERING DEPOSIT
(4-point loading)

TOP

Specimen No.	Strength ($X 10^3$ psi)
1	8.5
2	8.8
3	11.9
4	9.6
5	10.0
6	7.4
7	11.5
8	10.9
9	7.3
10	8.8
Avg	9.5 ± 1.6

BOTTOM

1	8.8
2	9.3
3	10.2
4	10.0
5	8.7
6	7.7
7	10.0
8	8.5
9	12.2
10	11.1
Avg	9.7 ± 1.3

incidence. Over the wavelength region 0.6 to 1.1 micrometers the transmittance shall be greater than 43%.

b) Absorption. The absorption over the 8-12 micrometer region shall be less than 0.01 per centimeter at 10.6 μm.

c) Parallelism. The provided blank shall have maximum allowable wedge of 10 minutes.

d) Strain. The distribution of permanent strain shall be symmetrical and the birefringence resulting from permanent strain shall not produce more than 10 nanometers relative retardation or path difference per centimeter of a transmitted narrow band light source.

e) Chips and fractures. A blank with a vented fracture exceeding 10 mm in length or aiming at the center of the blank will be rejected. Blanks having pressure or fire cracks deeper than 1 mm will be rejected. Other surface irregularities, pits or cracks will not extend into an envelope defined by the minimum allowable dimensions.

f) Scatter. The angular spread of a focused spot on a blank will not increase by more than 15 percent over the angular spread of the same spot without the sample in the beam over the wavelength region 0.6 to 1.2 micrometers. The angular spread over the wavelength region 8 to 12 micrometers will be less than 2 percent.

g) Identification and marking. Each blank will be individually bagged and the bag marked in accordance with MIL-STD-130 with the type of material, size, individual run number identification, and manufacturer's name or code symbol.

2.5.2 Deposition

A 327-hour zinc selenide deposit was conducted to yield the required engineering samples. The deposit was made using the 17 by 9 by 60 inch mandrel, similar to the Pre-Engineering deposit. Deposition conditions for this deposit are presented in Table 1.

Figure 7 displays the thickness profile of one of the plates from this deposit. Samples were located from the top of Plate C and fabricated into the specified blank dimensions. The overall optical quality of the zinc selenide was good. However, a portion of the material deposited late in the run contained a scattering layer. The layer did not interfere with locating the engineering samples and was outside the required thickness to produce a zinc selenide blank.

2.5.3 Tests on engineering samples

All twelve (12) engineering samples were tested and found to meet: dimensional requirements, specified chip and fracture criteria, and spectral transmittance requirements in the infrared and the visible. Typical transmittance curves are presented in Figures 8 and 9 for the infrared and visible spectra, respectively.

Engineering samples No. ENG-11 and ENG-12 were tested for absorption coefficient at 10.6 μm. Each sample was measured in three locations and the average absorption coefficient (including surface absorption) was determined. Absorption coefficients of $1.98 \times 10^{-3} cm^{-1}$ and $1.68 \times 10^{-3} cm^{-1}$ were measured for samples No. ENG-11 and ENG-12, respectively. The thickness of these two samples was 1.08 cm.

All twelve zinc selenide blanks tested for strain showed no relative retardation. Localized birefringence is present in individual crystallites; no larger order birefringence was discernible.

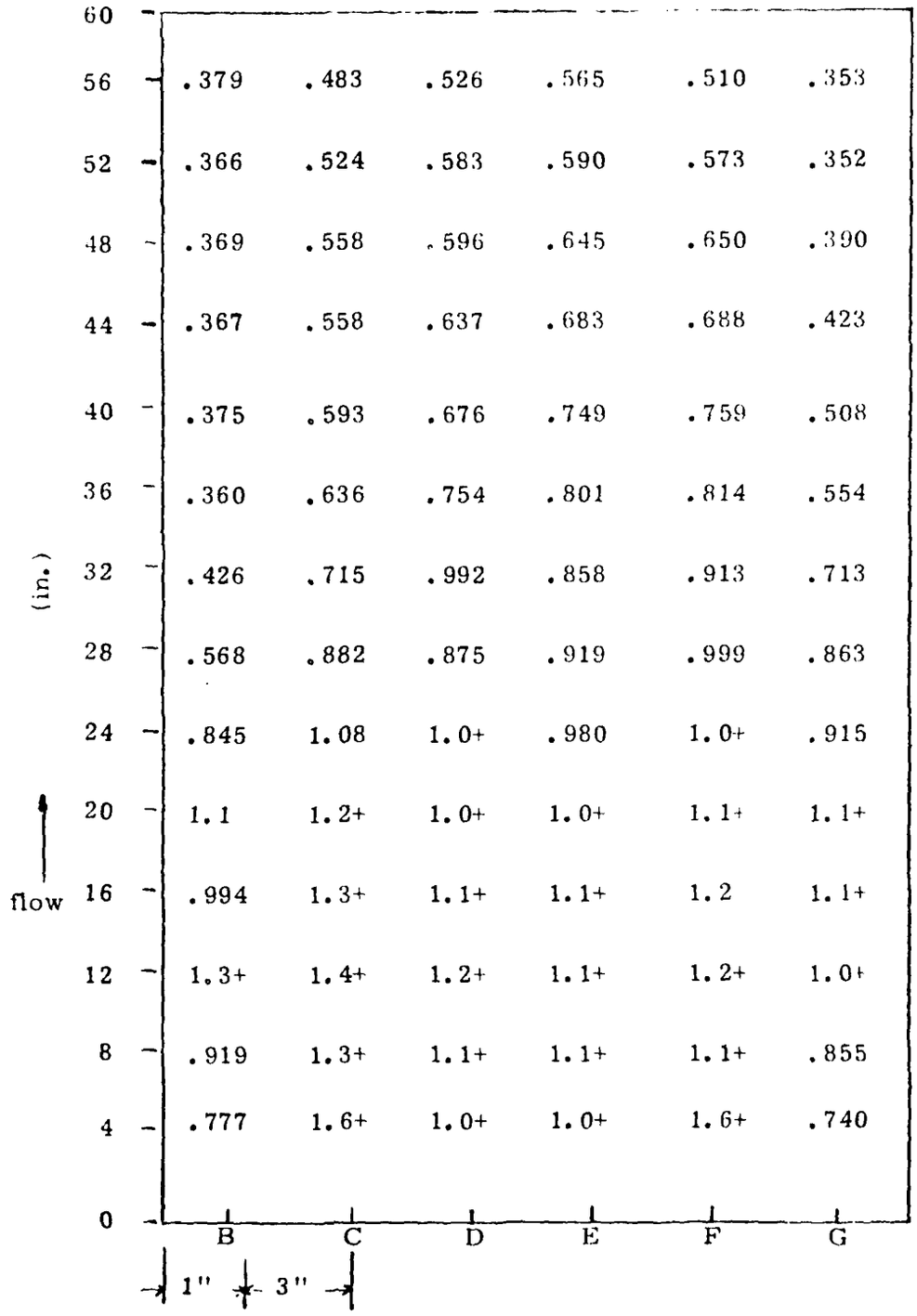

Figure 7. Thickness Profile, Plate A, Engineering Deposit.

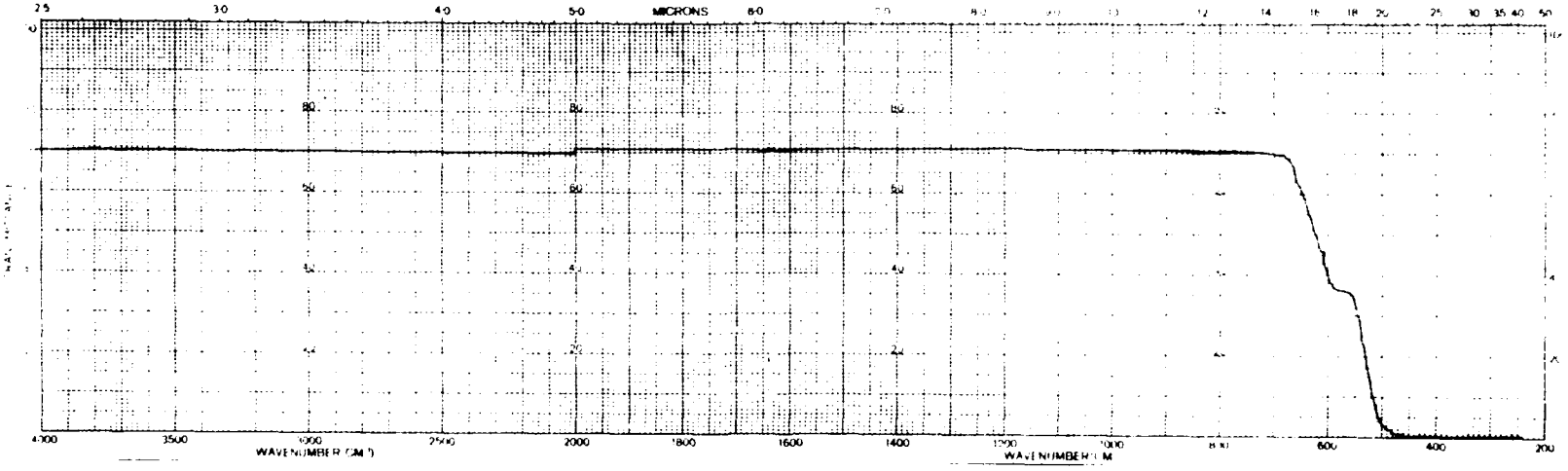

Figure 8. Infrared Transmittance of Sample ENG-1.

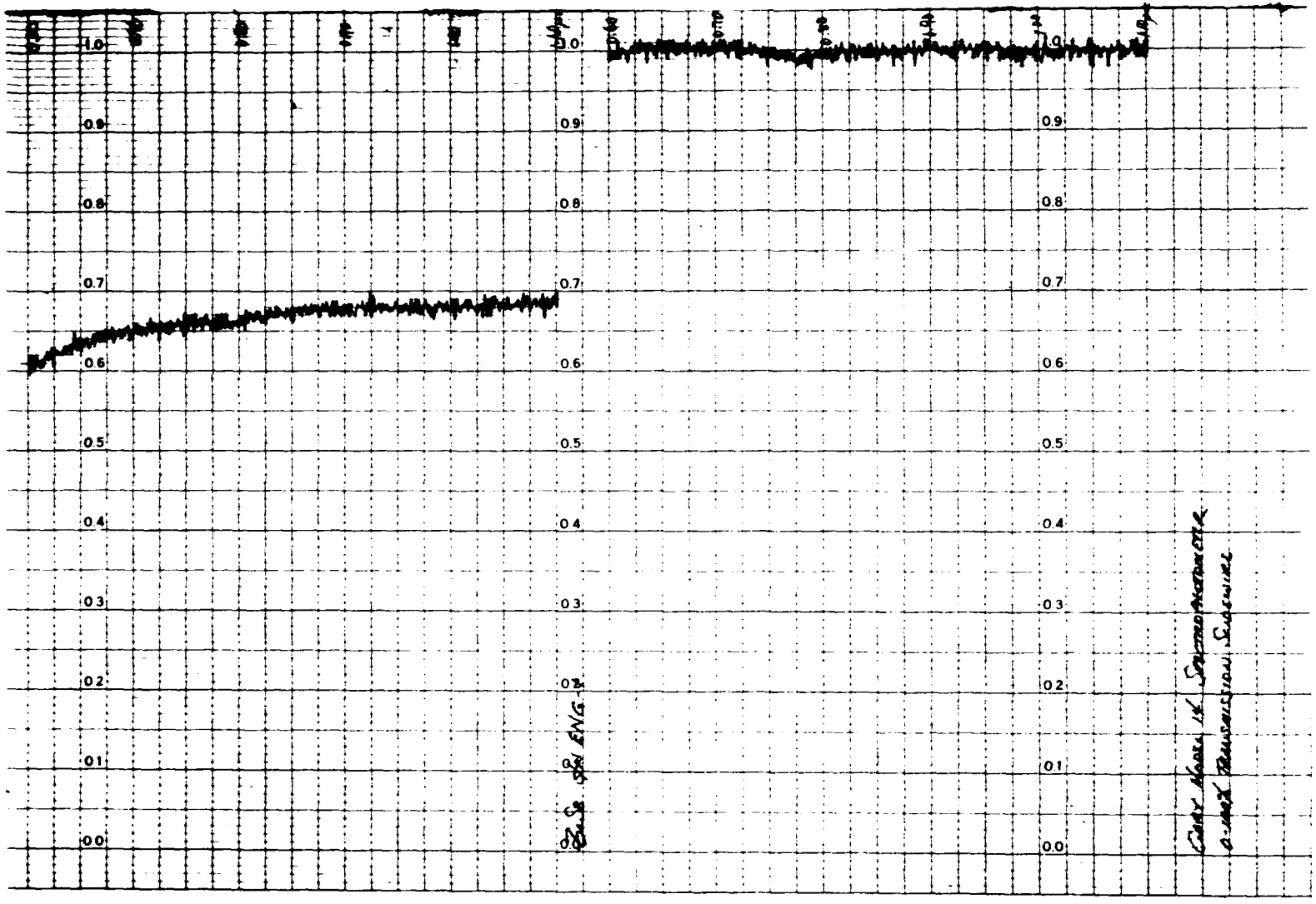

Figure 9. Transmittance From 0.6 to 1.1 μm of Sample ENG-1.

Table 3 presents the image spoiling data from samples No. ENG-11 and ENG-12. Sample No. ENG-12 did not meet the required image spoiling criteria of 2% image growth in the 8-12 μm range (3.1% tested for sample ENG-12). The samples were subsequently tested at the Night Vision and Electro-Optics Laboratory yielding the results shown in Table 4. Sample ENG-12 was measured at 2.8% image growth at 1/2 power for the 12 o'clock position, and 0% for the 3 o'clock position. This large variation with orientation indicates that a poor surface figure was the cause of the excessive image broadening. Interferograms of this sample revealed the nonuniformity of the surfaces. Sample ENG-12 was accepted as meeting the optical requirements of the contract.

2.6 Confirmatory Sample Deposit

2.6.1 Sample requirements

In this deposit it was a program requirement that the following blanks be fabricated: (20) lens blanks having sufficient thickness to yield the lens element as per drawing No. SM-C-804146 (Figure 10), (2) 49 mm diameter X 6.35 mm thick, (2) 61 mm diameter X 6.35 mm thick, and (2) 67.5 mm diameter X 6.35 mm thick. These blanks ot witness samples were tested to determine if they met the following specifications: (Description of the test procedures and test equipment are presented in the Appendix.)

a) Transmittance. The uncoated transmittance for a 6.35 mm thickness shall be greater than 58 percent over the wavelength region 8 to 13 μm at normal incidence. Over the wavelength region 0.6 to 1.1 μm the transmittance shall be greater than 43 percent.

b) Inclusions. The maximum size inclusion is 0.625 mm. The permissible number of maximum size inclusion is one per each cubic centimeter of material. The sum of the diameters of all inclusions in any given cubic centimeter of material shall not exceed 0.615 mm. Bubbles are classified as inclusions.

TABLE 3

IMAGE SPOILING DATA

Sample No.	Image Width (μrad) No. Sample	With Sample	% Image Growth	Spectra
ENG-11	155.7	158.6	1.8	8-12 μm
ENG-12	155.7	160.5	3.1	
ENG-11	16.8	17.9	6.5	0.6-1.1 μm
ENG-12	16.8	17.9	6.5	

TABLE 4

IMAGE SPOILING DATA

(Measured at Night Vision Laboratory)

	Visible ½ Power	1/e Power	I R ½ Power	1/e Power
ENG Sample 11 oriented at 12 o'clock				
Reg A	1.7%	6.2%	1.4%	1.9%
Reg B	1.7%	3.8%		
ENG Sample 11 oriented at 3 o'clock				
Reg A	4.5%	7.8%	1.8%	1.8%
Reg B	1.3%	4.2%		
ENG Sample 12 oriented at 12 o'clock				
Reg A	2.6%	6.8%	2.8%	1.3%
Reg B	0.3%	1.8%		
ENG Sample 12 oriented at 3 o'clock				
Reg A	3.9%	7.2%	0%	1.8%
Reg B	0.1%	0.8%		

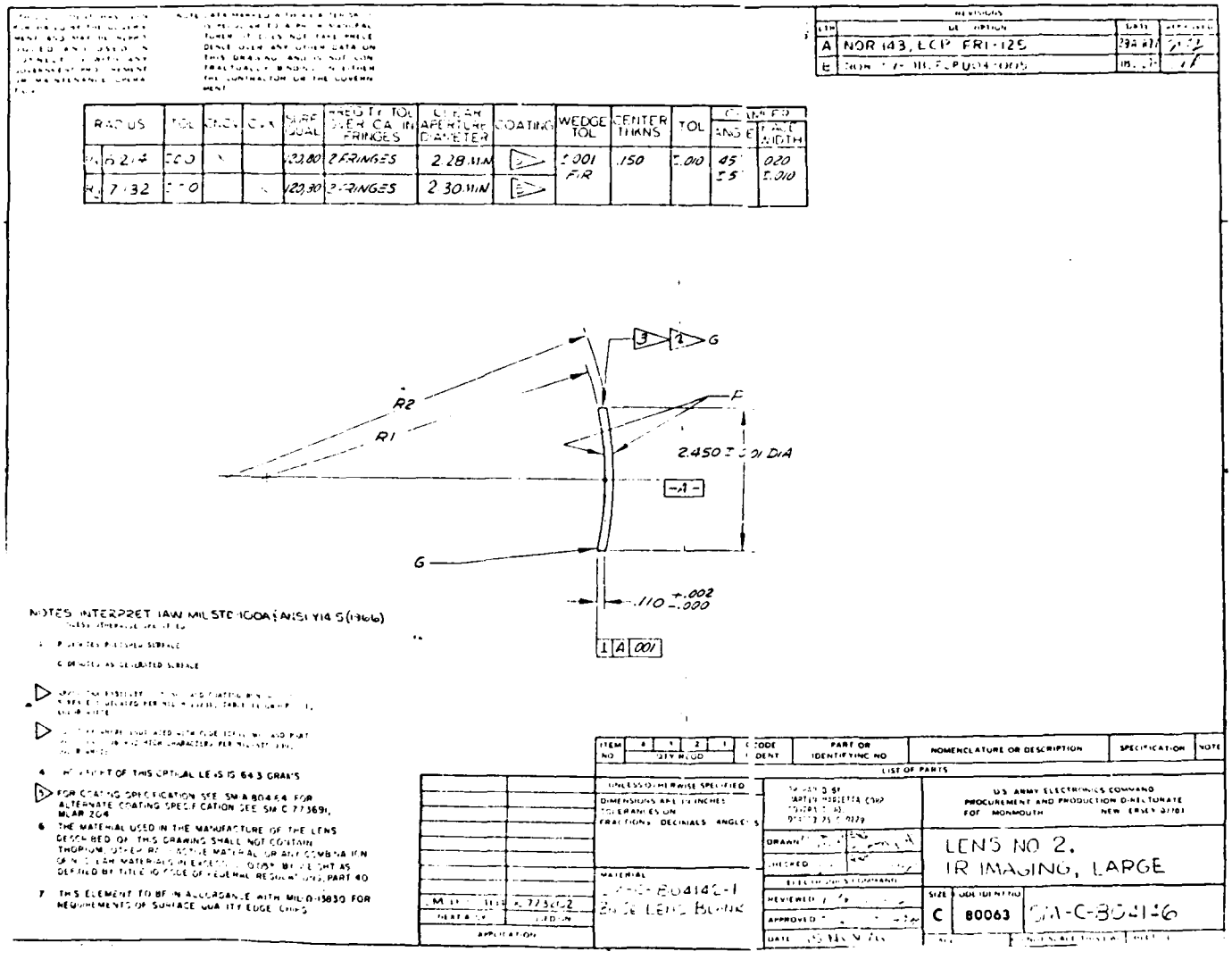

Figure10 . Zinc Selenide Lens Blank.

c) Surface Hardness. The Knoop, 50 gram, hardness rating shall be at least 100.

d) Absorption. The absorption over the 8 to 12 micrometer region will be less than 0.01 per centimeter. The absorption at 10.6 micrometers will be less than 0.005 per centimeter.

e) Scatter. The angular spread of a focused spot on a blank 6.35 mm thick shall increase by no more than 15 percent over the angular spread of the same spot without the sample in the beam over the wavelength region 0.6 to 1.2 micrometers. The angular spread over the wavelength region 8 to 12 micrometers will be less than 2 percent.

f) Rupture Modulus. The modulus of rupture shall average 7300 pounds per square inch with a minimum value of not less than 6570 psi.

g) Parallelism. The provided blanks shall have maximum allowable wedge of 10 minutes. The blank(s) used for image spoiling tests will have a maximum wedge of 0.5 minute.

h) Strain. The distribution of permanent strain shall be symmetrical, and the birefringence resulting from permanent strain will not produce more than 10 nanometers relative retardation of path difference per centimeter of a transmittance narrow-band light source.

i) Chips and Fractures. A blank with a vented fracture exceeding 10 mm in length or aiming at the center of the blank shall be rejected. Blanks having pressure or fire cracks deeper than 1 mm shall be rejected. Other surface irregularities, pits, or cracks shall not extend into 2.55 mm diameter of the blanks required to yield the lens, as per drawing No. SM-C-804146 (Figure 10).

2.6.2 Substrate modifications

An alternative method of depositing zinc selenide on a curved substrate rather than on a flat was incorporated into the Confirmatory deposit. In this method, the zinc selenide is deposited close to the final geometry of the lens on one side. Figure 11 displays the concept of the curved mandrel. This method of producing lenses shortens the deposition time and results in a direct cost reduction. Further, it should reduce the fabrication cost of polishing the lens to its final dimensions. New curved substrates were fabricated for use in the confirmatory deposit and the pilot run.

2.6.3 Deposition

A 143 hour zinc selenide deposit was conducted to yield the required confirmatory samples. (Table 1 presents the deposition conditions for this deposit.) The deposit was made in a graphite box mandrel of inside dimensions 12 X 22 X 63 inches. The thickness profile for one of the deposited plates is presented in Figure 12 revealing adequate overall thickness and uniformity. Also shown are the locations of the concave lens sites and the thickness of the lens blanks.

The zinc vaporization system performed well throughout the 143 hour deposit. No malfunctions of any kind were detected.

2.6.4 Test results on confirmatory samples

The six (6) polished samples from the confirmation deposit were tested and found to meet: the dimensional requirements, specified chip and fracture criteria, and spectral transmittance required in the infrared and the visible spectra. Typical transmittance curves for the polished samples are presented in Figures 13 and 14 showing the infrared and visible spectra, respectively.

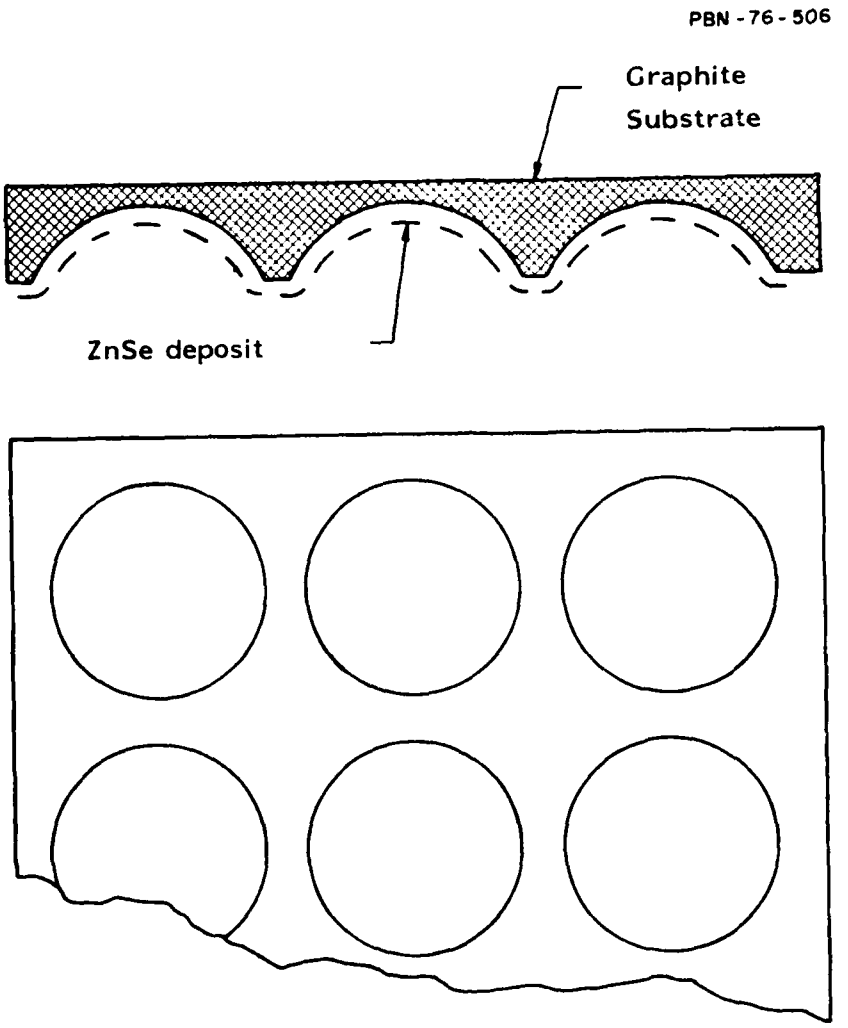

Figure 11. Curved Mandrel Used in Confirmatory and Pilot Deposits.

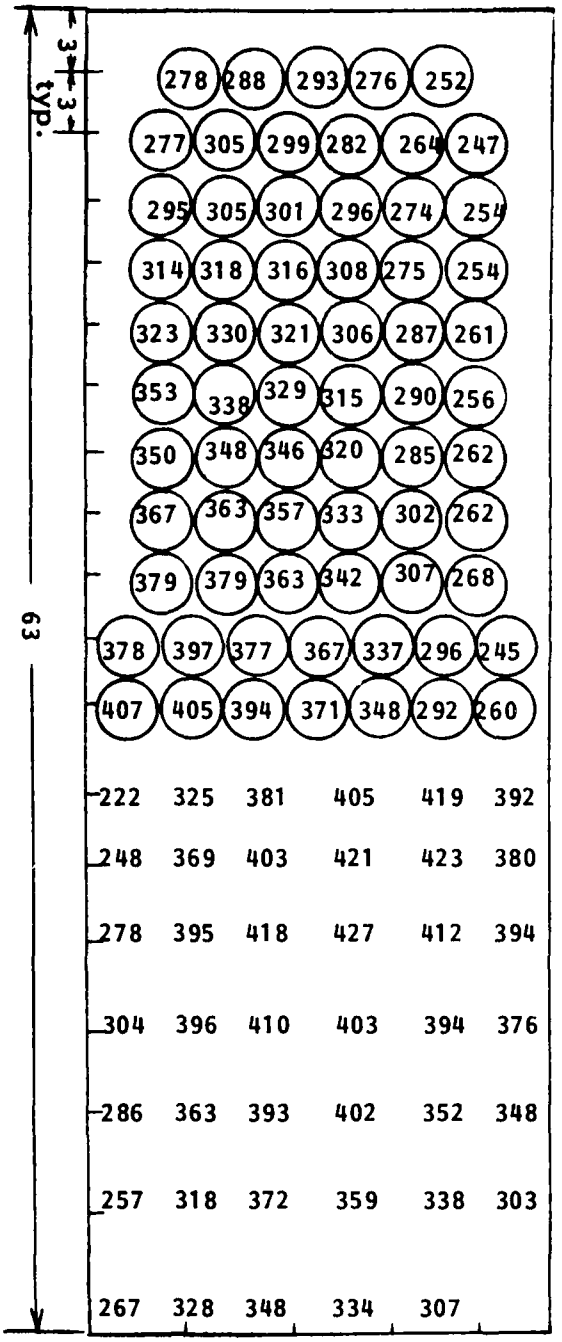

Figure 12. Thickness Profile, Plate A, Confirmatory Deposit.

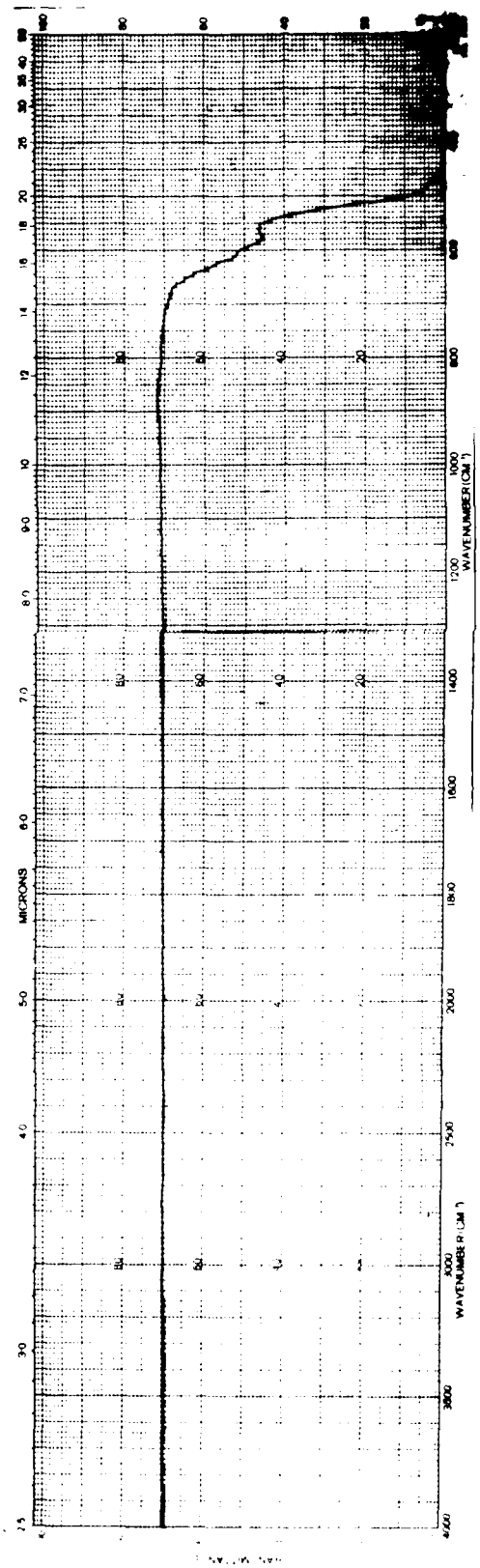

Figure 13. Infrared Transmittance, Sample CON No. 1.

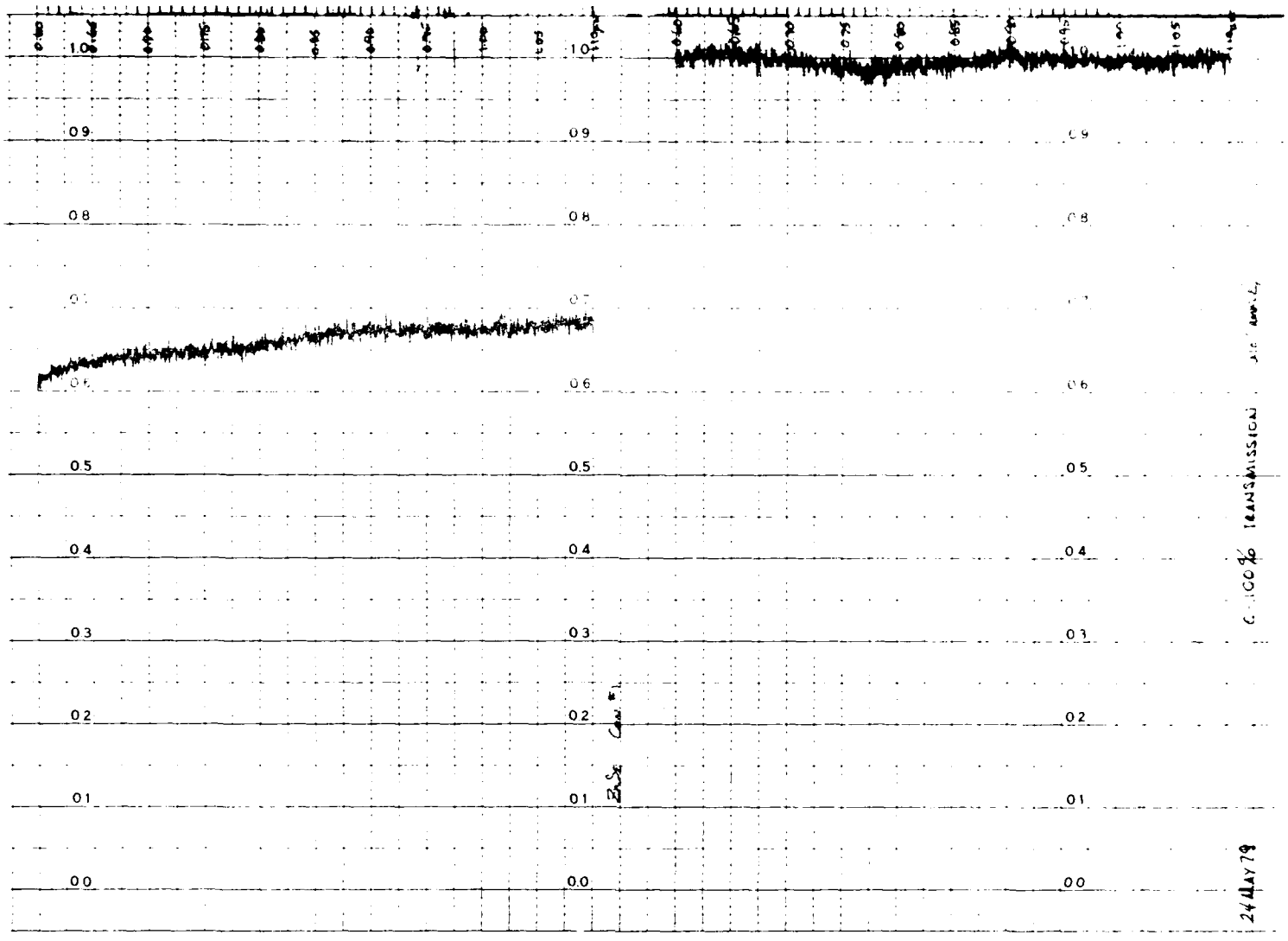

Figure 14. Visible Transmittance, Sample CON No. 1.

The absorption at 10.6 micrometers measured for each of the polished samples is presented in Table 5. The measured values of approximately 0.002 cm^{-1} are were within the required specification. Four of the six polished samples were tested for image spoiling characteristics in the 0.6-1.1 micrometer and the 8-12 micrometer bands. This data is presented in Table 6.

Zinc selenide from the top and bottom sections of the confirmatory deposit was fabricated into samples for modulus of rupture testing. Table 7 presents the results. All samples tested met the minimum strength requirements of 6570 psi, and the ten samples tested exceed the required 7300 psi average modulus of rupture.

The six polished test blanks tested for strain showed no relative retardation. Some localized birefringence was seen in individual crystallites; no larger order birefringence was discernible.

2.7 Pilot Production Run

2.7.1 Production rate capability

The production rate capability of producing 481 zinc selenide lens blanks (largest diameter of 75 mm) per month was demonstrated in the pilot run. A graphite box mandrel, 12 X 22 X 60 inches, was used for the deposition. Approximately 400 lens sites are available in this size mandrel: the two 12 X 60 inch plates each contain approximately 60 sites, and the larger 22 X 60 inch plates contain approximately 120 sites each.

The pilot run was of 108 hours duration. Two deposits of similar duration can easily be deposited every month, yielding a total of 800 possible lens blanks per month. Typical efficiencies of 75 percent, for out of furnace yield, and 80 percent yield for cutting and coring, will produce the required monthly rate. Actual yields for this particular pilot run were close to 100 percent for the curved mandrel sites yielding lens blanks. Two percent of the lens blanks were lost during the cutting operations.

TABLE 5

ABSORPTION COEFFICIENT @ 10.6 μm FOR CONFIRMATORY DEPOSIT

Specimen No.	Absorption Coefficient (cm^{-1})
CON 1	0.0023
CON 2	0.0023
CON 3	0.0022
CON 4	0.0023
CON 5	0.0020
CON 6	0.0021

*Includes surface absorption

TABLE 6

IMAGE SPOILING DATA - CONFIRMATORY DEPOSIT

Sample No.	Image Width @ 50% Intensity (μrad) No Sample	With Sample	% Image Growth
	8-12 μm		
CON #3	201.3	206.8	2.7
CON #4	195.1	198.6	1.8
CON #5	198.0	196.6	---
CON #6	204.5	202.4	---
	0.6328 μm		
CON #3	19.2	17.7	---
CON #4	15.7	16.8	7.0
CON #5	15.7	17.7	12.7
CON #6	15.7	17.3	10.2

TABLE 7

MODULUS OF RUPTURE DATA FOR CONFIRMATORY DEPOSIT

4-Point Loading

TOP

Sample No.	Strength (psi X 10^3)
1	9.57
2	9.60
3	9.27
4	8.11
5	6.61

BOTTOM

1	8.49
2	6.91
3	6.71
4	7.18
5	8.87

Avg. 8.13 ± 1.2

2.7.2 Sample requirements

For the pilot run it was a program requirement that the following blanks be fabricated: (40) lens blanks having sufficient thickness to yield the lens element as per drawing No. SM-C-804146 (Figure 10), (4) 49 mm diameter X 13 mm thick, (4) 61 mm diameter X 13 mm thick, and (4) 67.5 mm diameter X 13 mm thick. These blanks or witness samples were tested to determine if they met the following specifications. (Description of the test procedures and test equipment are presented in the Appendix.)

a) Transmittance. The uncoated transmittance for a 6.35 mm thickness shall be greater than 58 percent over the wavelength region 8 to 13 micrometers at normal incidence. Over the wavelength region 0.6 to 1.1 micrometers the transmittance shall be greater than 43 percent.

b) Inclusions. The maximum size inclusion is 0.625 mm. The permissible number of maximum size inclusions is one per each cubic centimeter of material. The sum of the diameters of all inclusions in any given cubic centimeter of material shall not exceed 0.625 mm. Bubbles are classified as inclusions.

c) Surface Hardness. The Knoop, 50 gram, hardness rating shall be at least 100.

d) Absorption. The absorption over the 8 to 12 micrometer region will be less than 0.01 per centimeter. The absorption at 10.6 micrometers will be less than 0.005 per centimeter.

e) Scatter. The angular spread of a focused spot on a blank 6.35 mm thick shall increase by more than 15 percent over the angular spread of the same spot without the sample in the beam over the wavelength region 0.6 to 1.2 micrometers. The angular spread over the wavelength region 8 to 12 micrometers will be less than 2 percent.

f) Rupture Modulus. The modulus of rupture shall average 7300 pounds per square inch with a minimum value of not less than 6570 psi.

g) Parallelism. The provided blanks shall have maximum allowable wedge of 10 minutes. The blank(s) used for image spoiling tests will have a maximum wedge of 0.5 minute.

h) Strain. The distribution of permanent strain shall be symmetrical, and the birefringence resulting from permanent strain will not produce more than 10 nanometers relative retardation or path difference per centimeter of a transmitted narrow-band light source.

i) Chips and Fractures. A blank with a vented fracture exceed 10 mm in length or aiming at the center of the blank shall be rejected. Blanks having pressure or fire cracks deeper than 1 mm shall be rejected. Other surface irregularities, pits, or cracks shall not extend into 2.55 mm diameter of the blanks required to yield the lens, as per drawing No. SM-C-804146.

2.7.3 Test results on Pilot run

Twelve blanks from the Pilot run [(4) 40 mm diameter, (4) 61 mm diameter, (4) 67.5 mm diameter)] were polished and tested for optical properties. Figures 15 through 26 display the spectral transmittance in the 0.6 to 1.1 micrometer range, while Figures 27 through 38 present the transmittance in the 8 to 12 micrometer range. All samples tested meet the uncoated transmittance requirements.

Table 8 presents the absorption coefficient at 10.6 micrometer measured for each of the twelve samples. The absorption coefficient for each of the twelve is less than the value required in the specification. Eight of the twelve polished samples were tested for image spoiling characteristics at 0.6328 micrometer and in the 8 to 12 micrometer range. The resultant data is shown in Table 9 and as noted all samples meet the required specification.

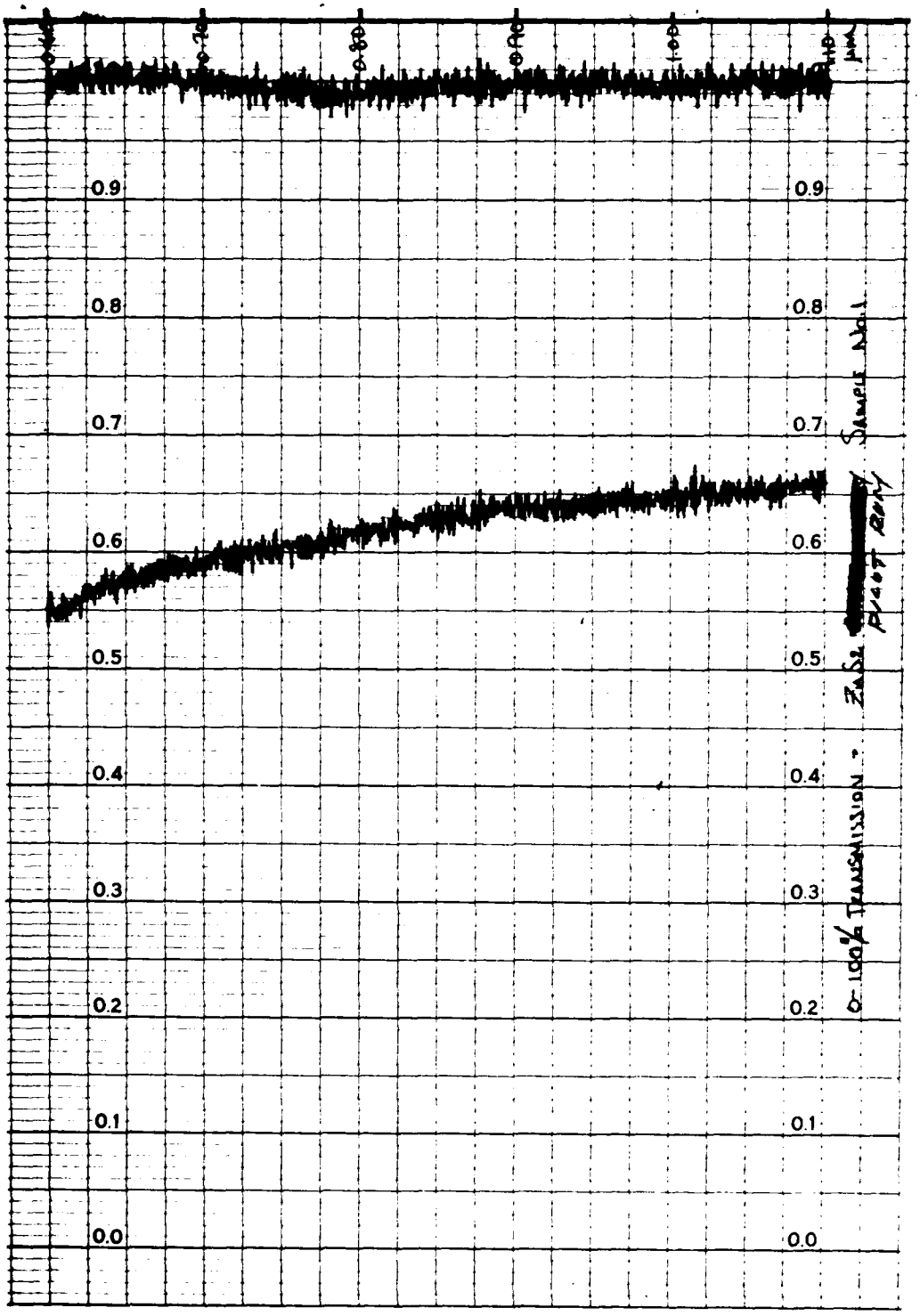

Figure 15. Visible Transmittance, Sample No. 1, Pilot Run.

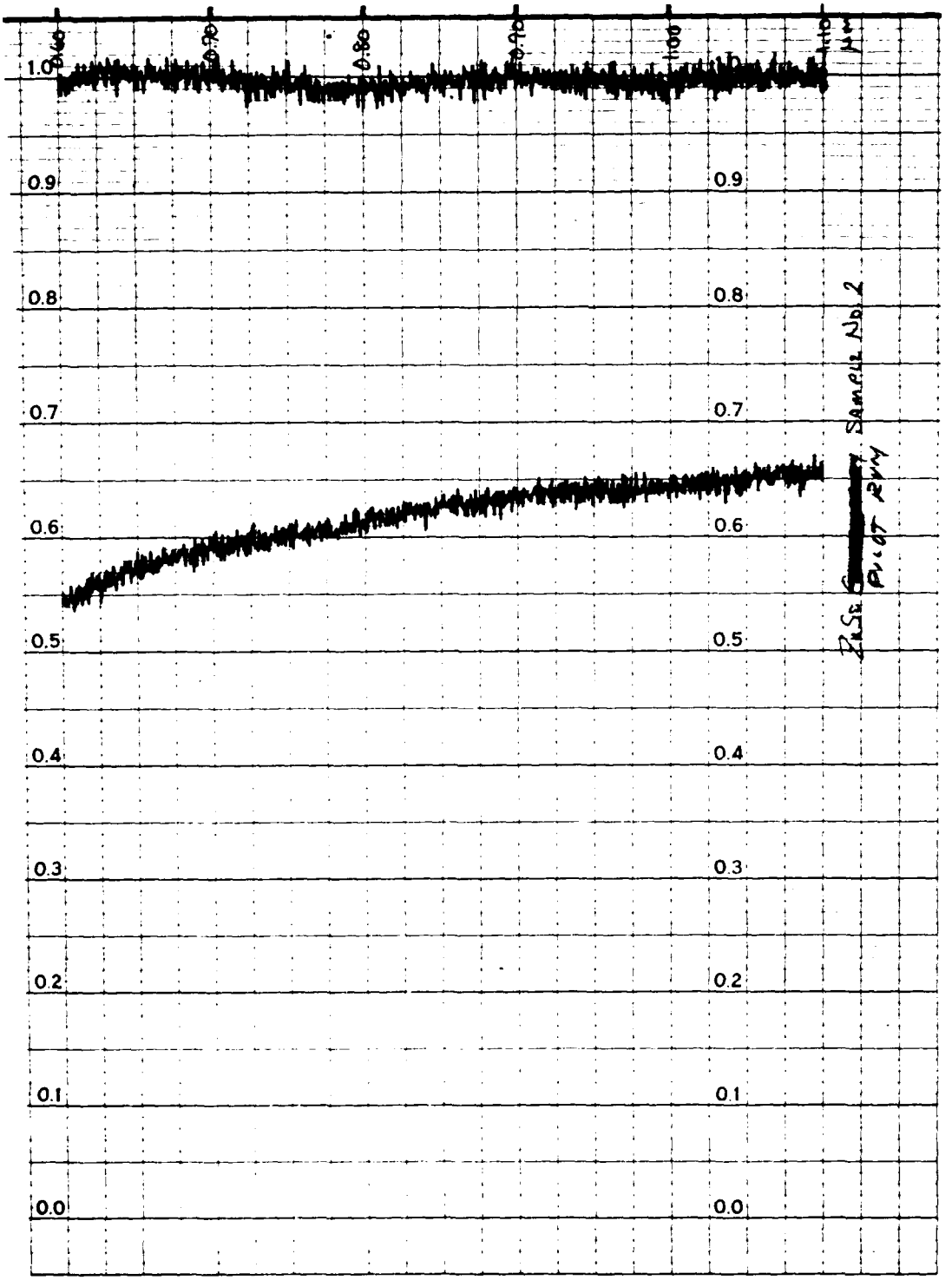

Figure 16. Visible Transmittance, Sample No. 2, Pilot Run

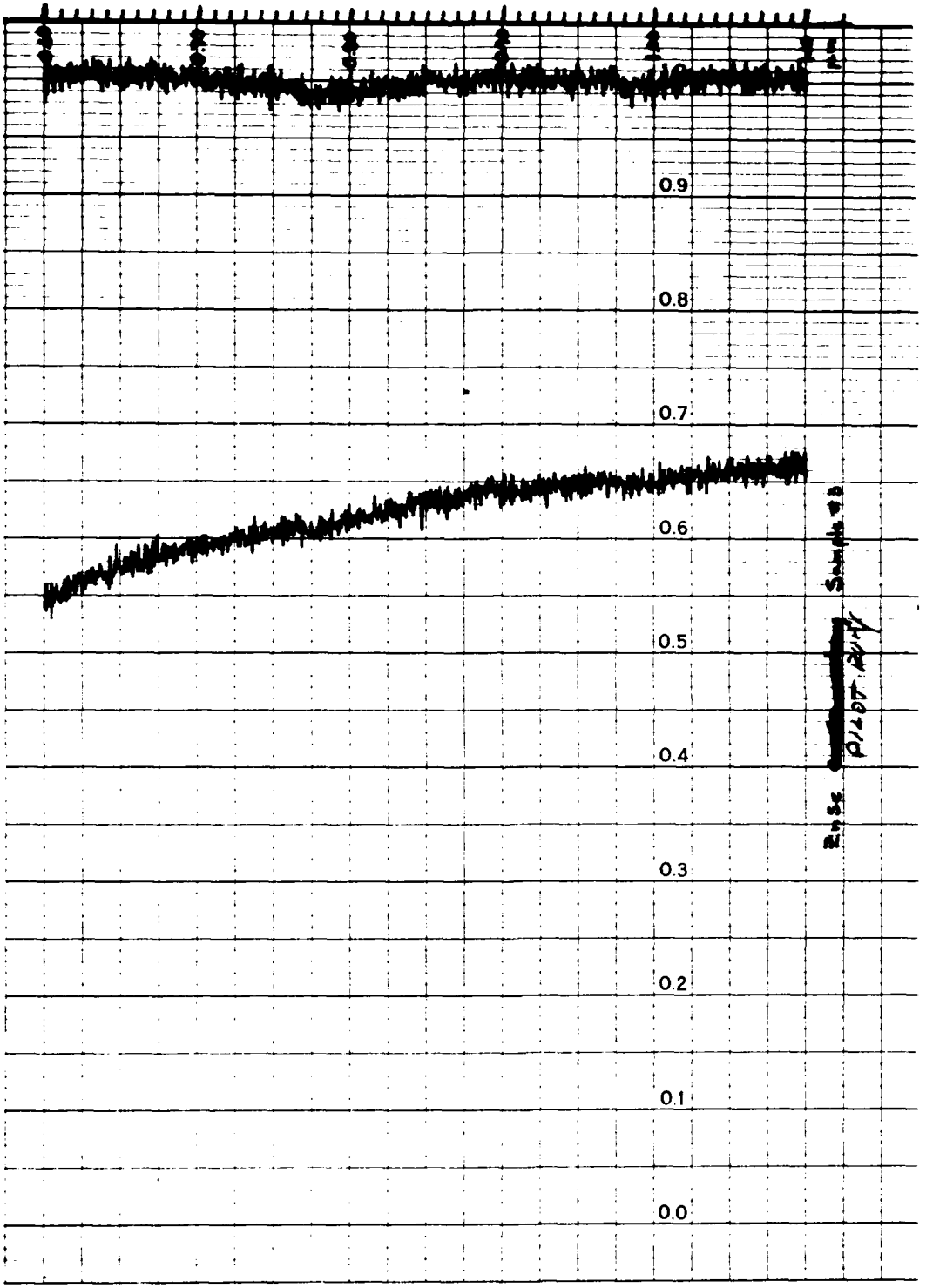

Figure 17. Visible Transmittance, Sample No. 3, Pilot Run

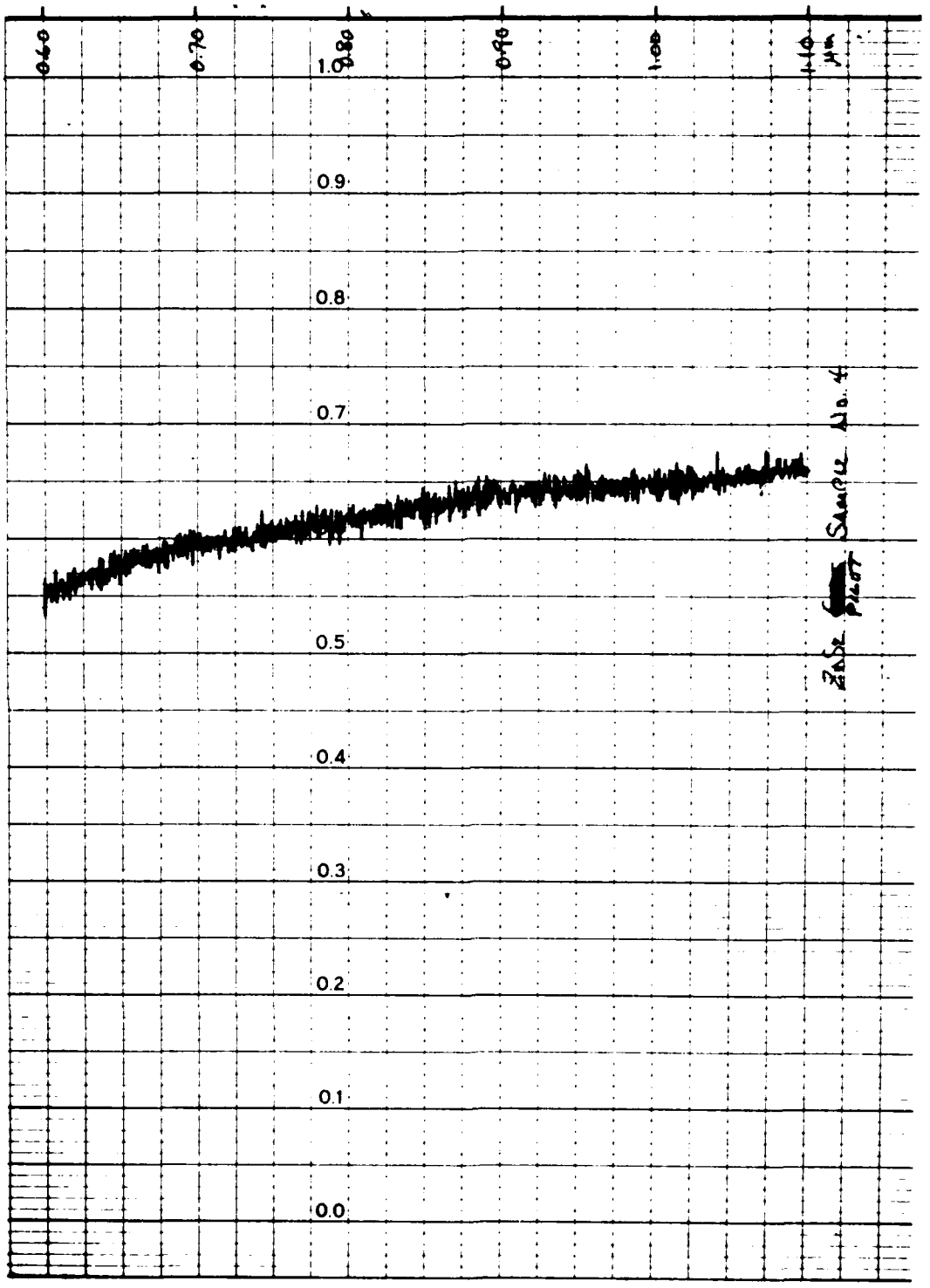

Figure 18. Visible Transmittance, Sample No. 4, Pilot Run

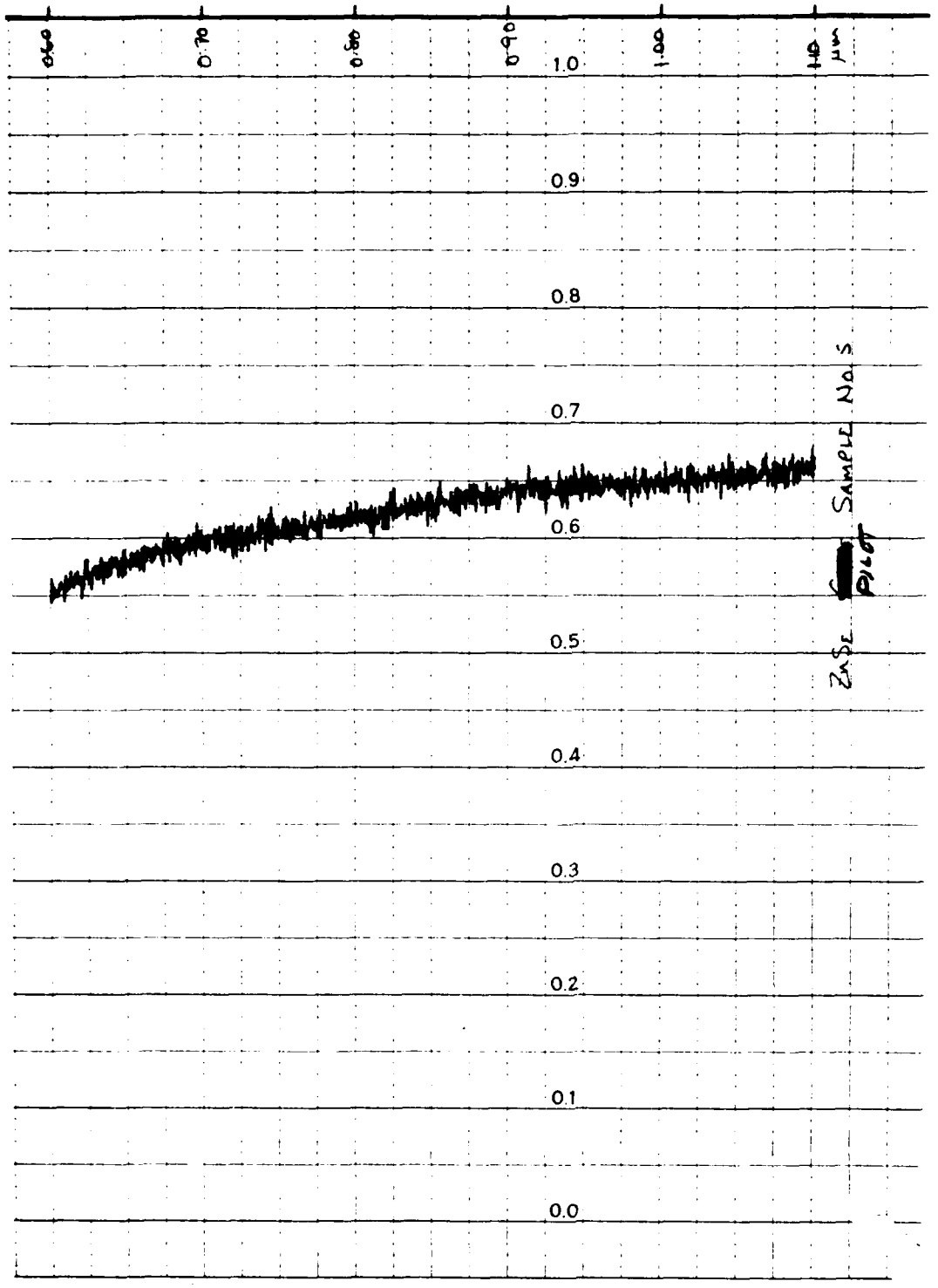

Figure 19. Visible Transmittance, Sample No. 5, Pilot Run

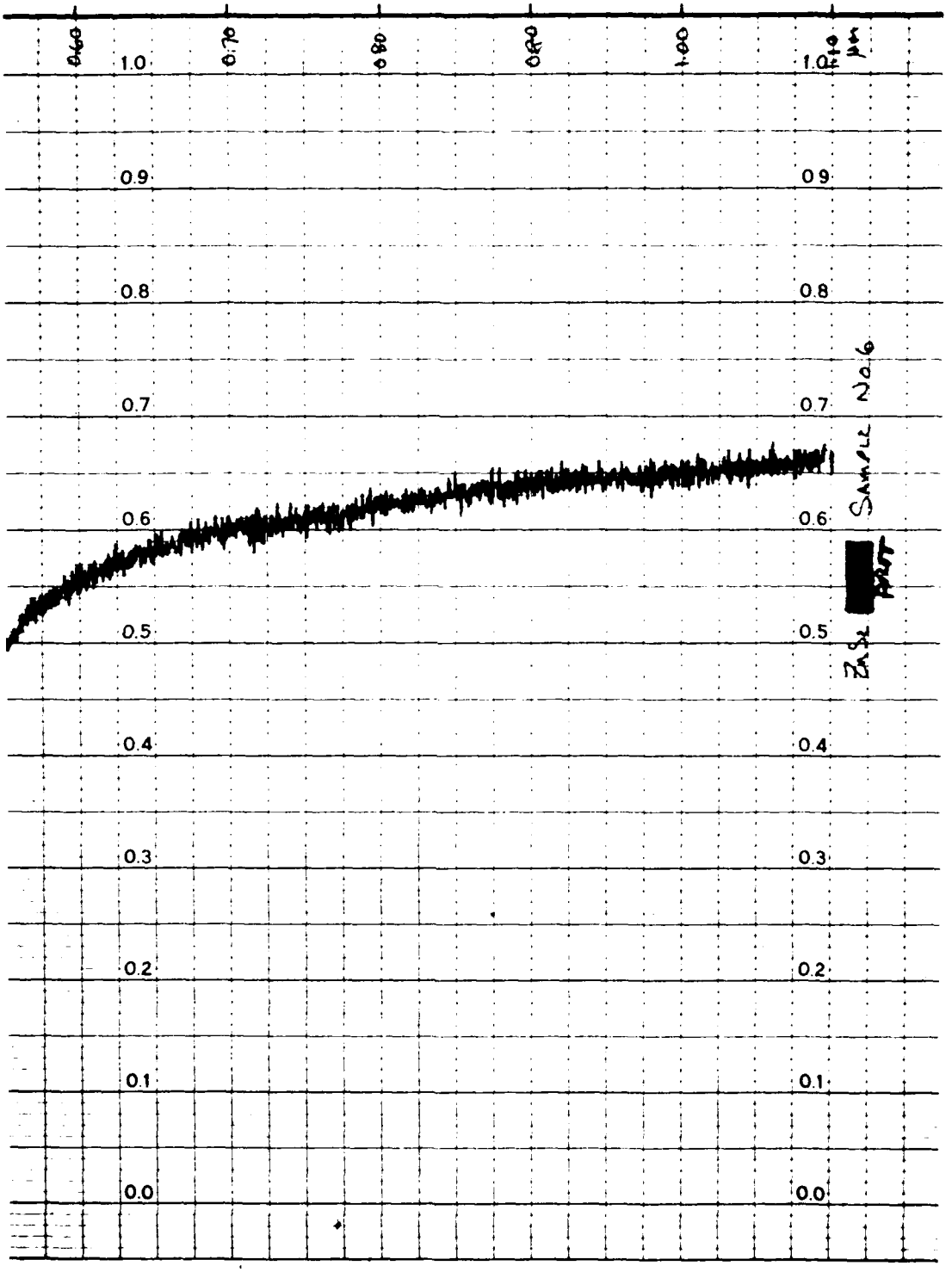

Figure 20. Visible Transmittance, Sample No. 6, Pilot Run

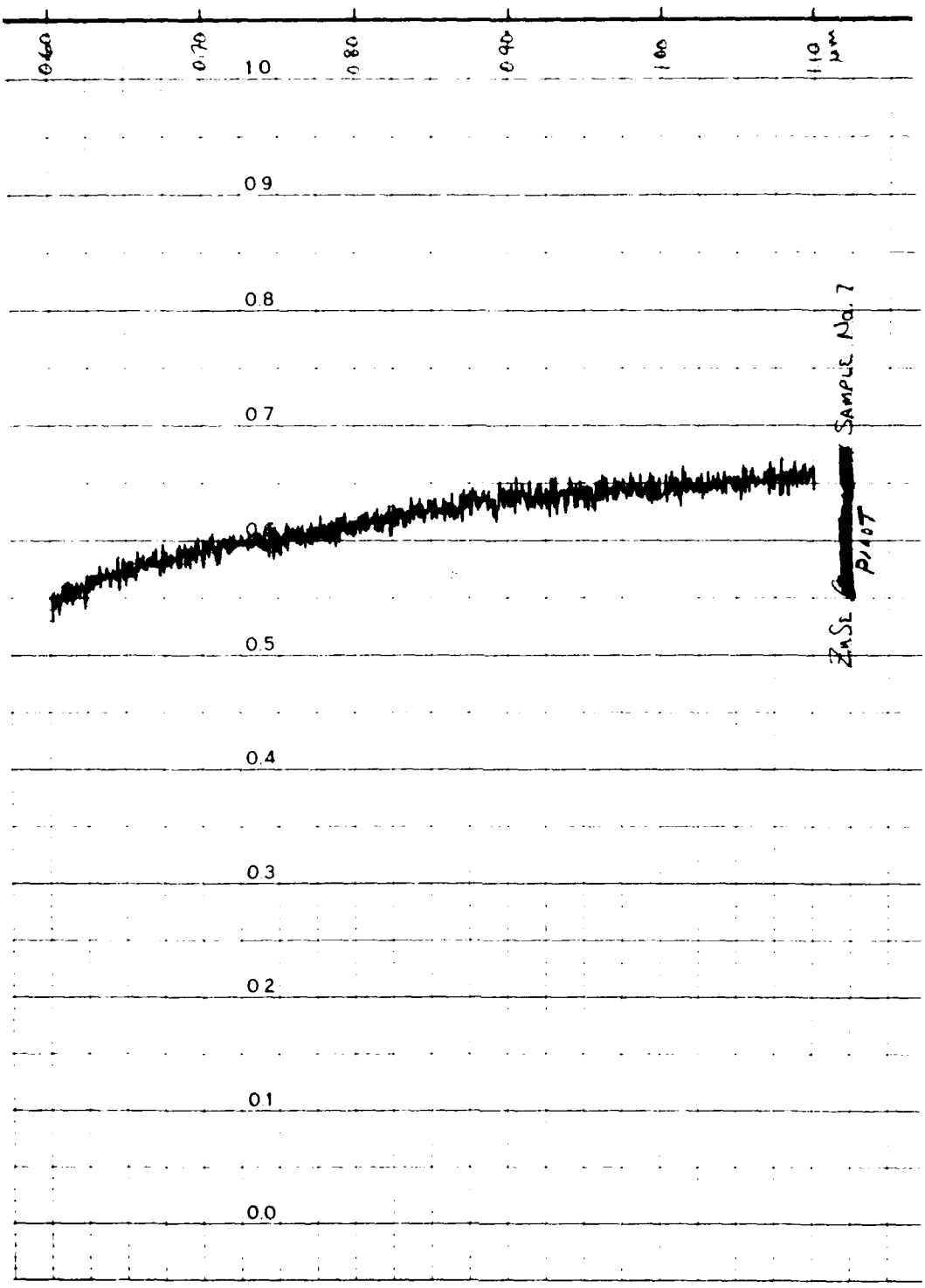

Figure 21 Visible Transmittance, Sample No. 7, Pilot Run

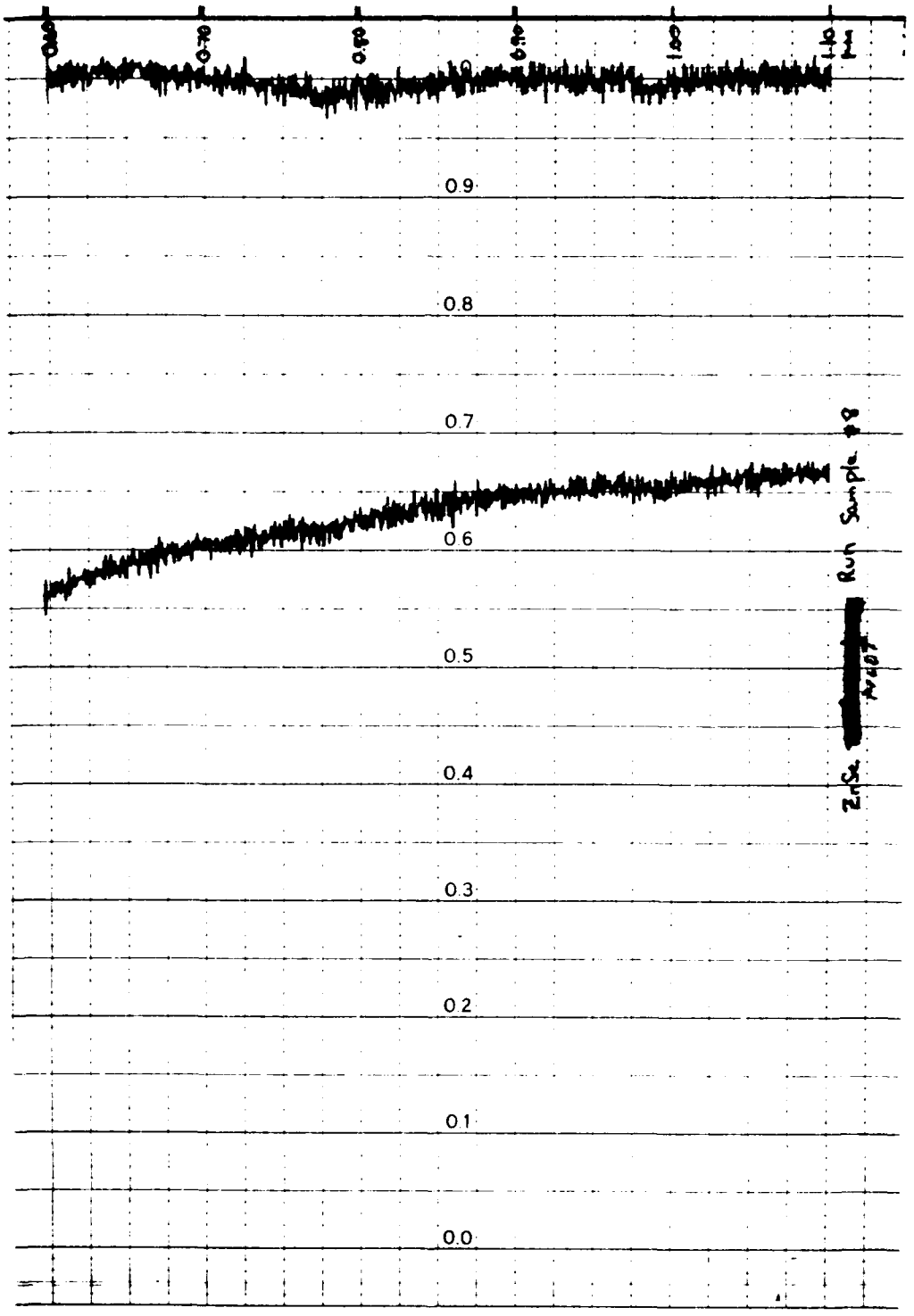

Figure 22. Visible Transmittance, Sample No. 8, Pilot Run

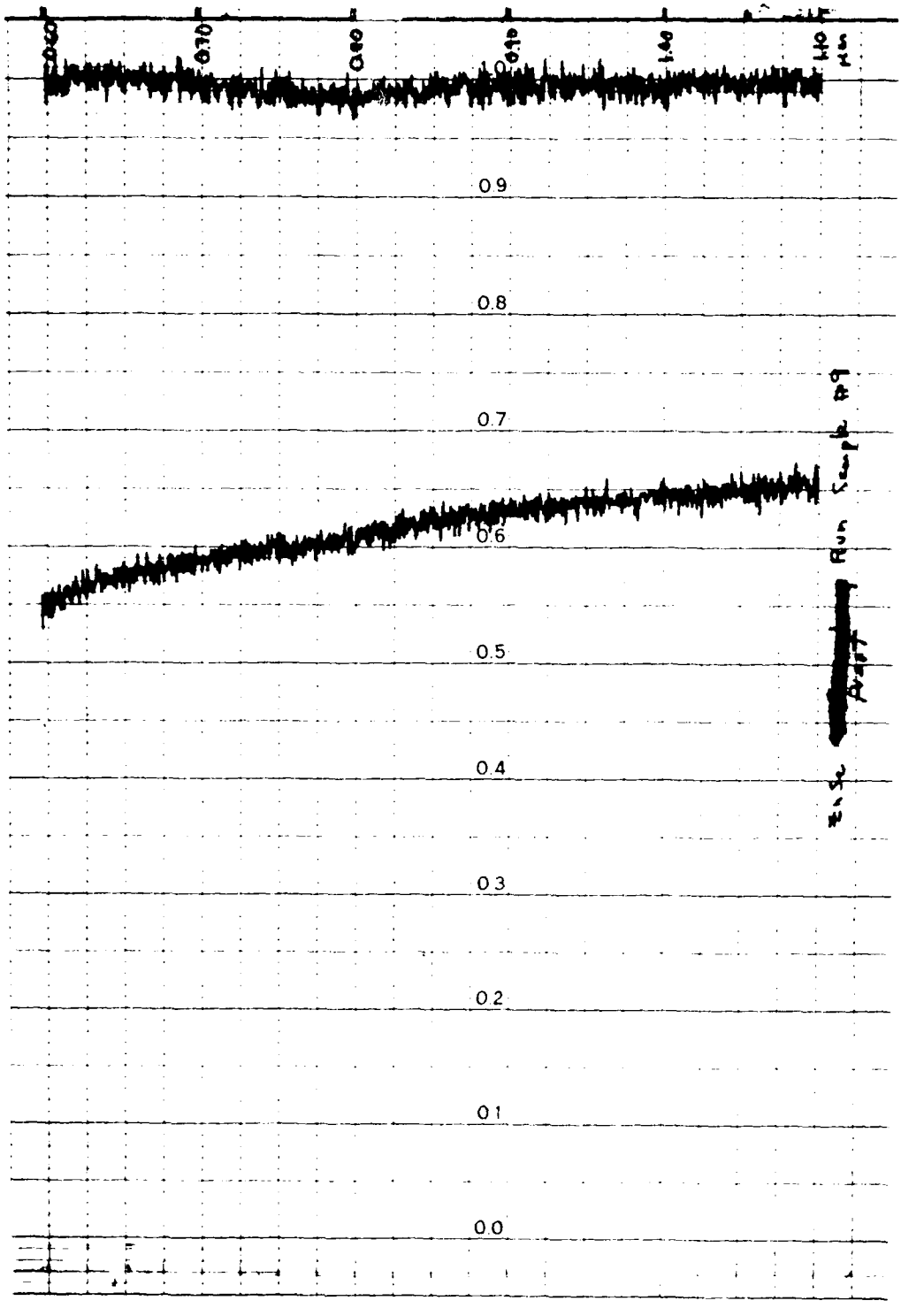

Figure 23. Visible Transmittance, Sample No. 9, Pilot Run

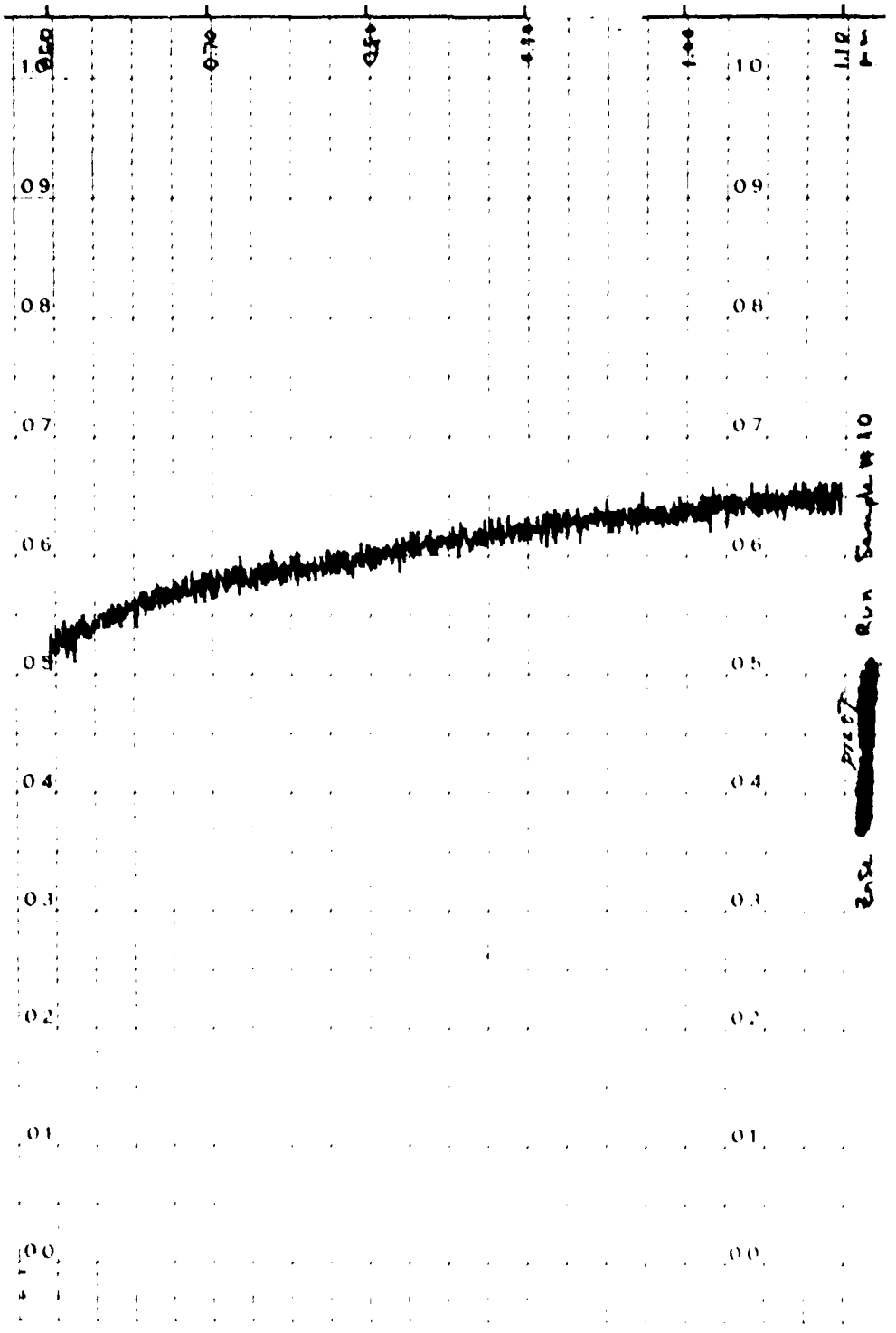

Figure 29. Visible Transmittance, Sample No. 10, Pilot Run

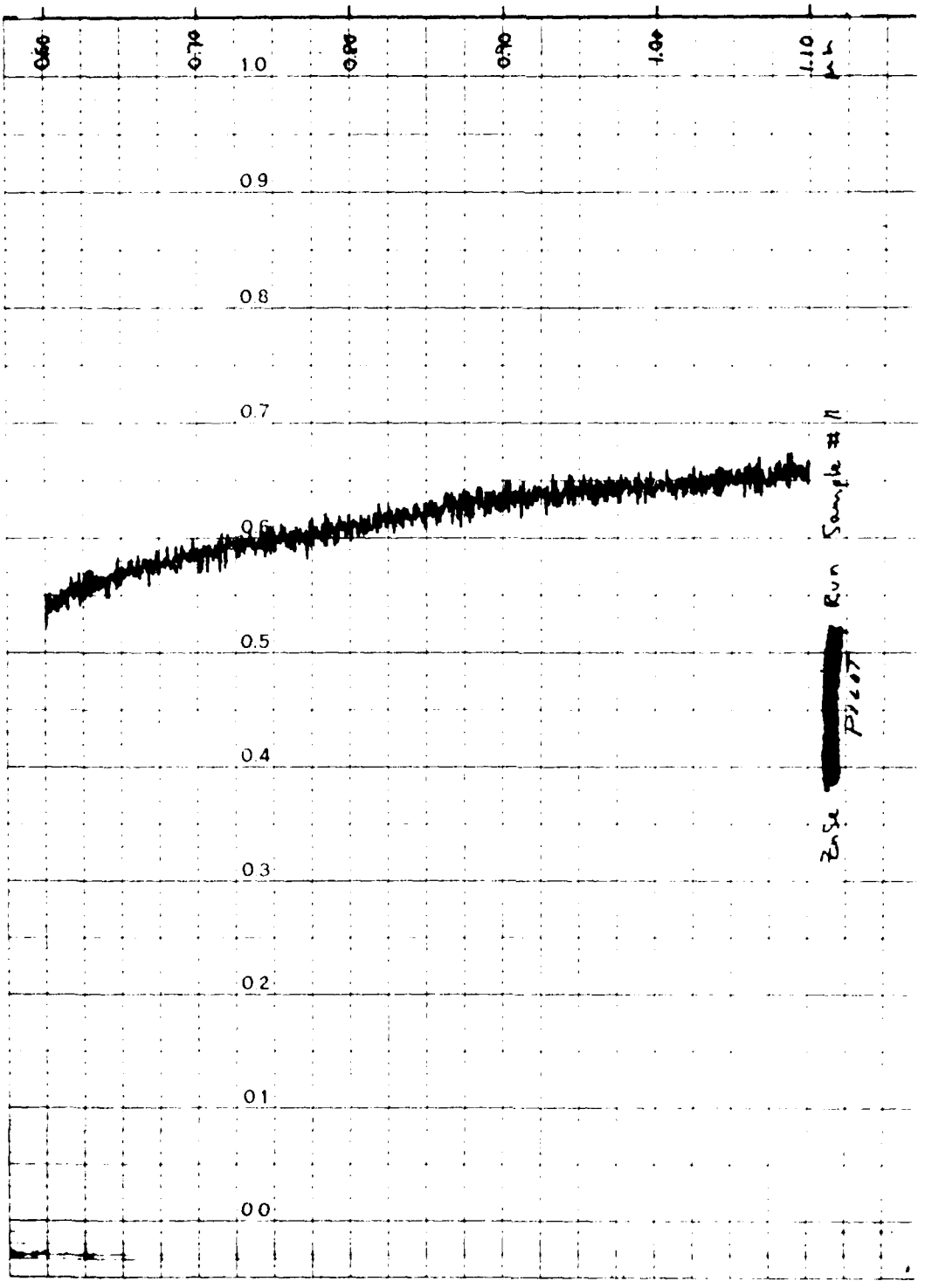

Figure 25. Visible Transmittance, Sample No. 11, Pilot Run

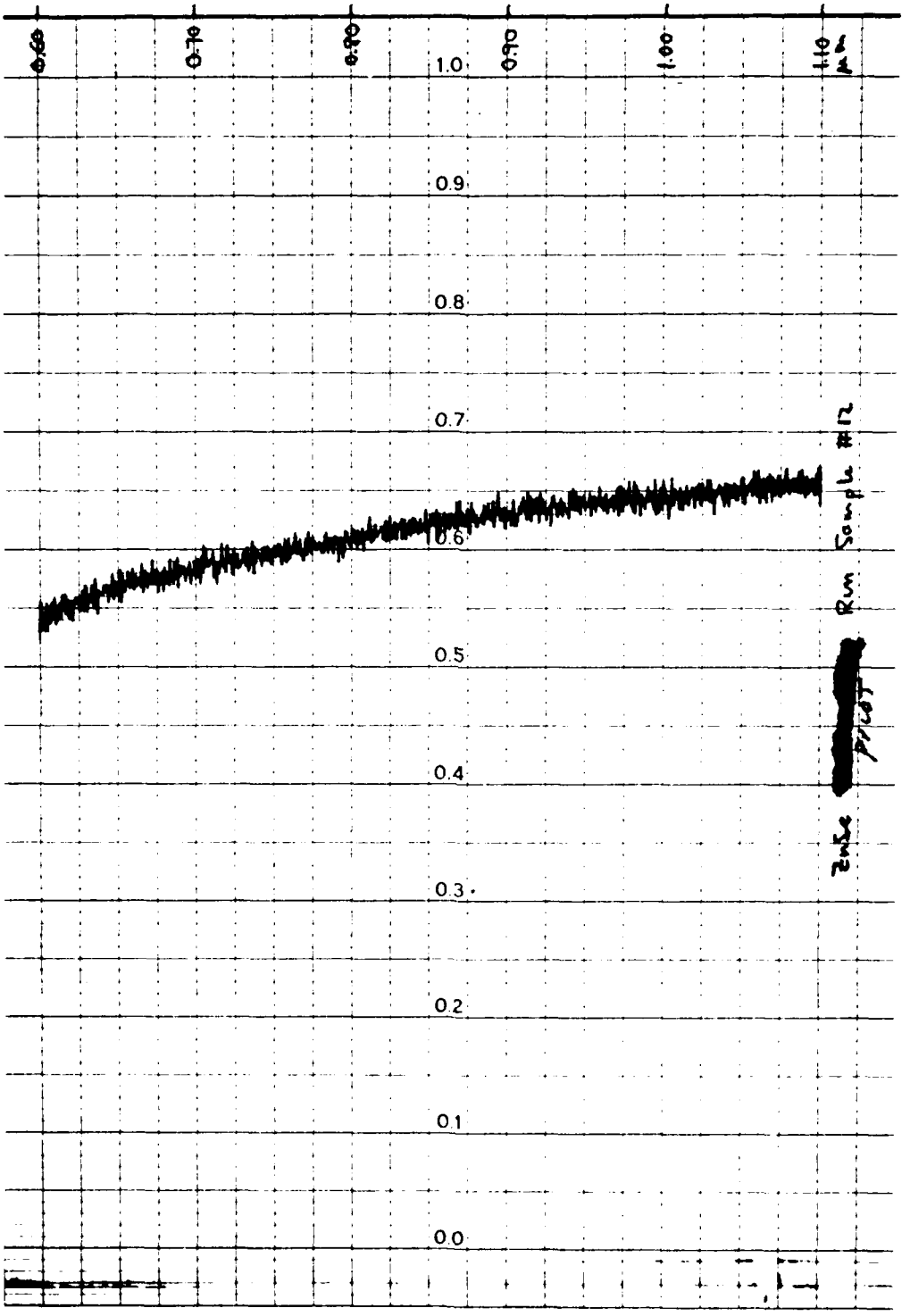

Figure 26. Visible Transmittance, Sample No. 12, Pilot Run

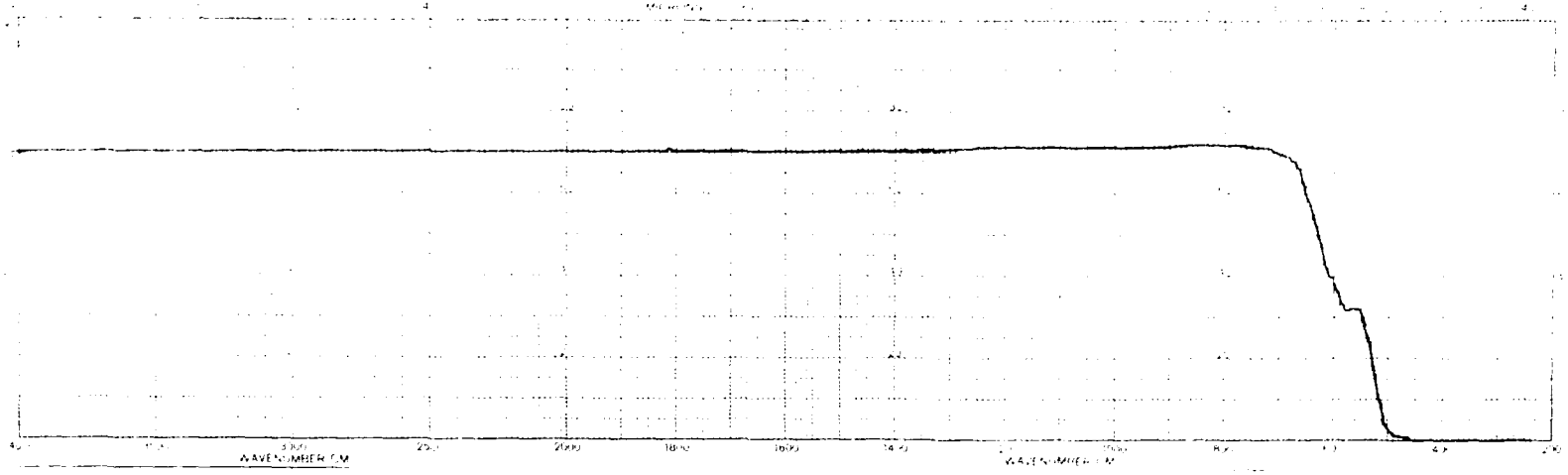

Figure 27. Infrared Transmittance, Sample No. 1, Pilot Run

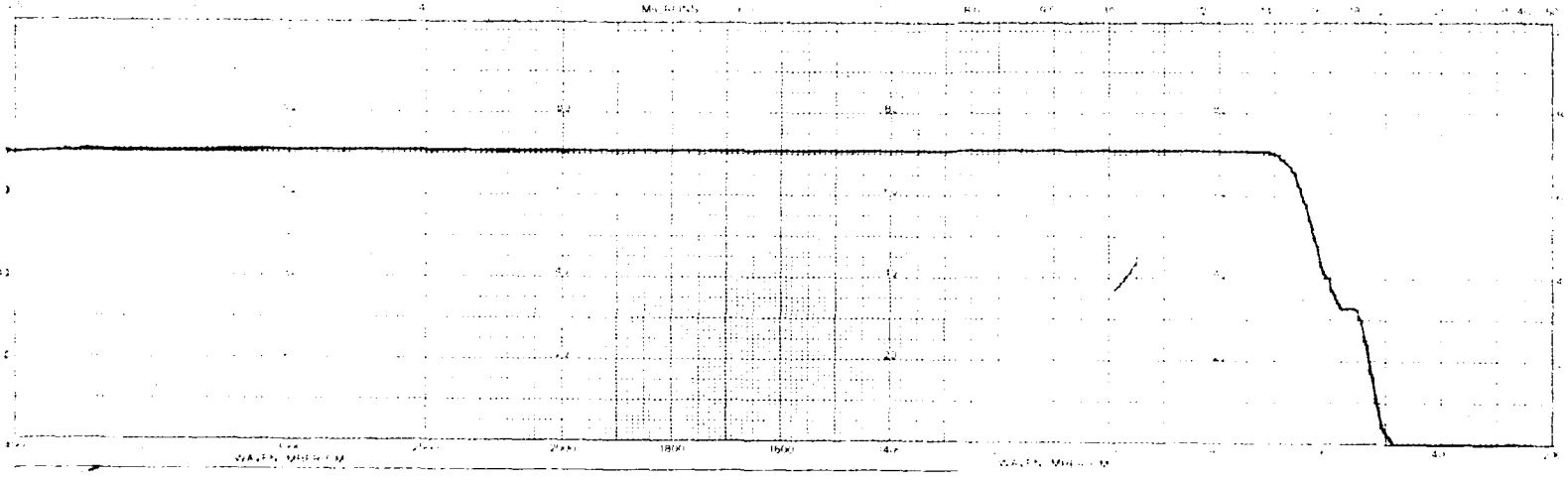

Figure 28. Infrared Transmittance, Sample No. 2, Pilot Run

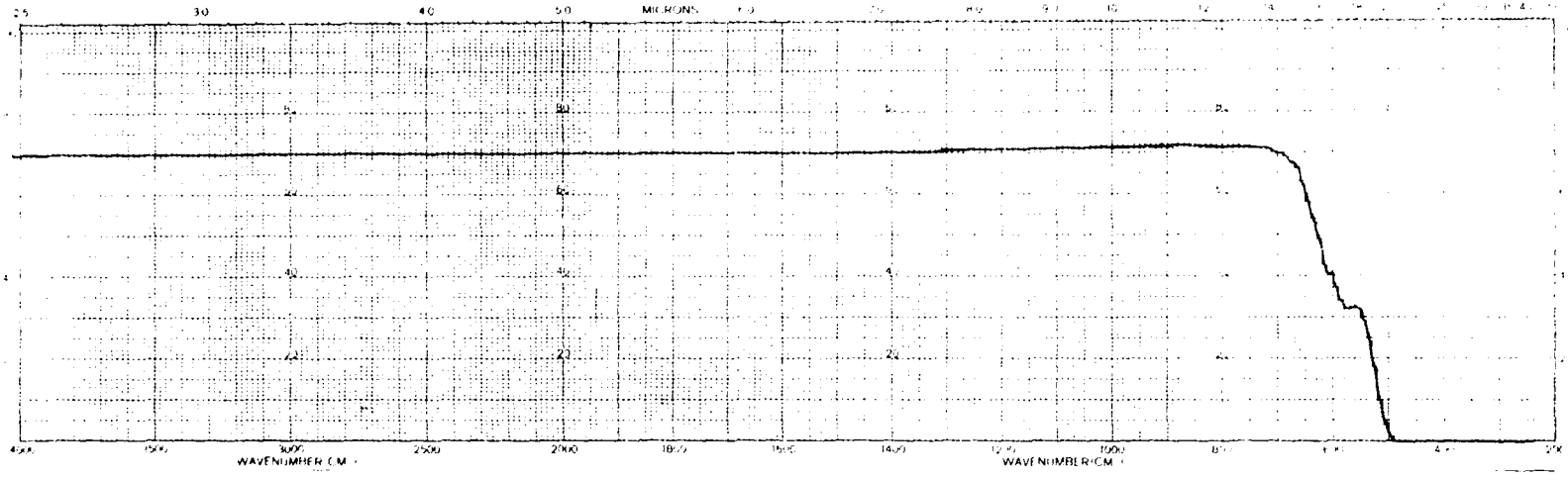

Figure 29. Infrared Transmittance, Sample No. 3, Pilot Run.

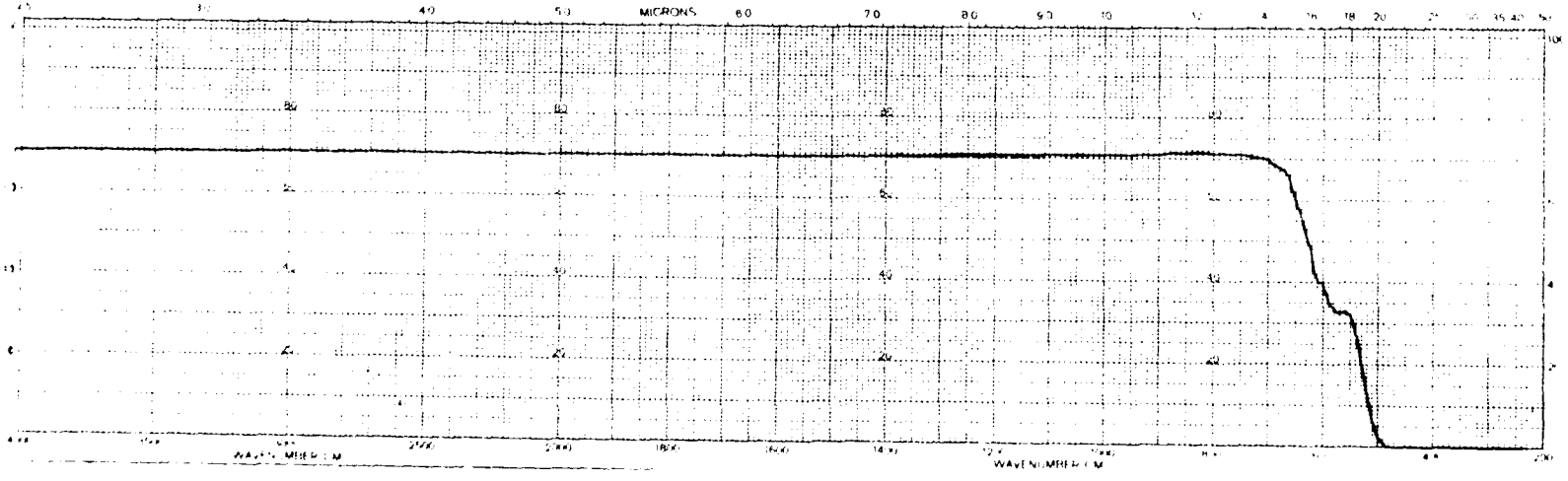

Figure 30. Infrared Transmittance, Sample No. 4, Pilot Run.

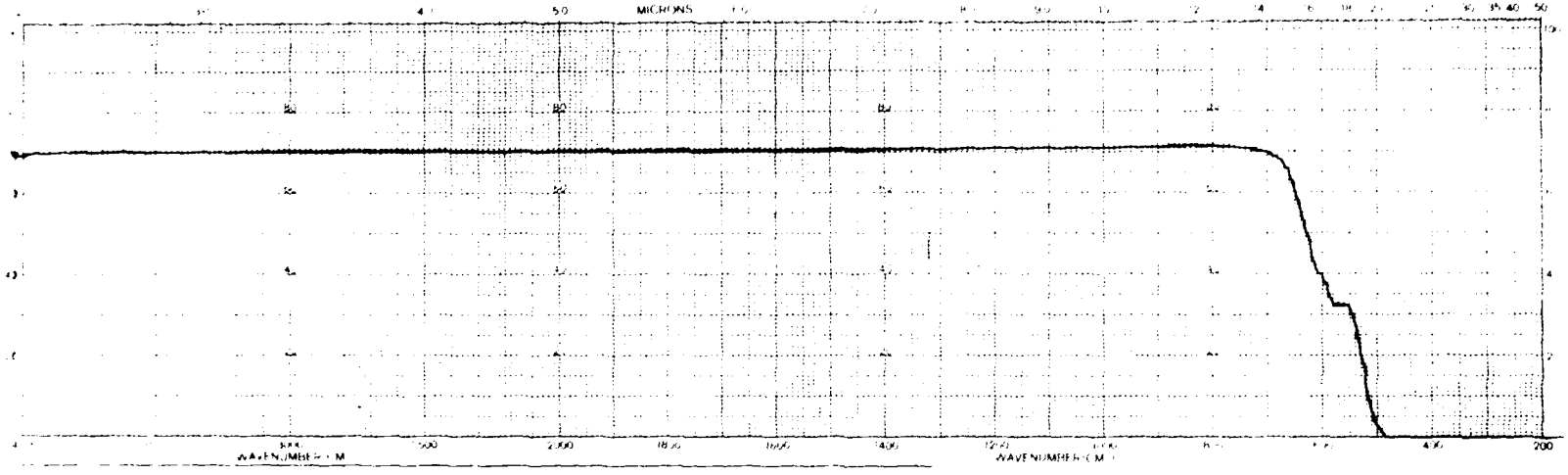

Figure 31. Infrared Transmittance, Sample No. 5, Pilot Run.

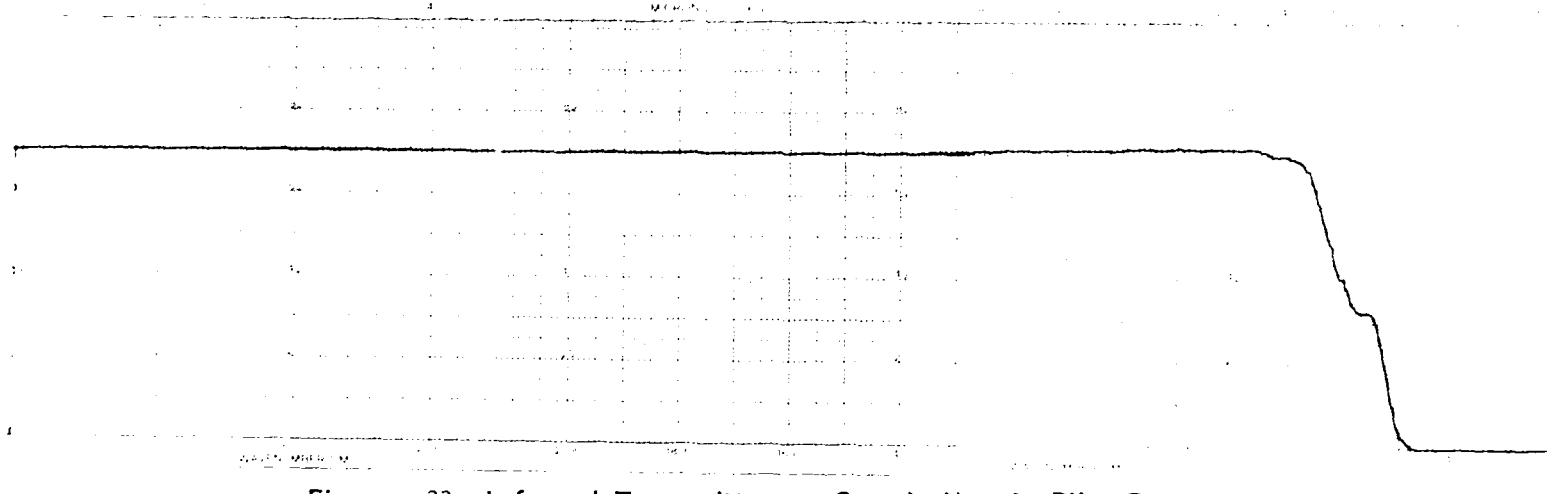

Figure 32. Infrared Transmittance, Sample No. 6, Pilot Run.

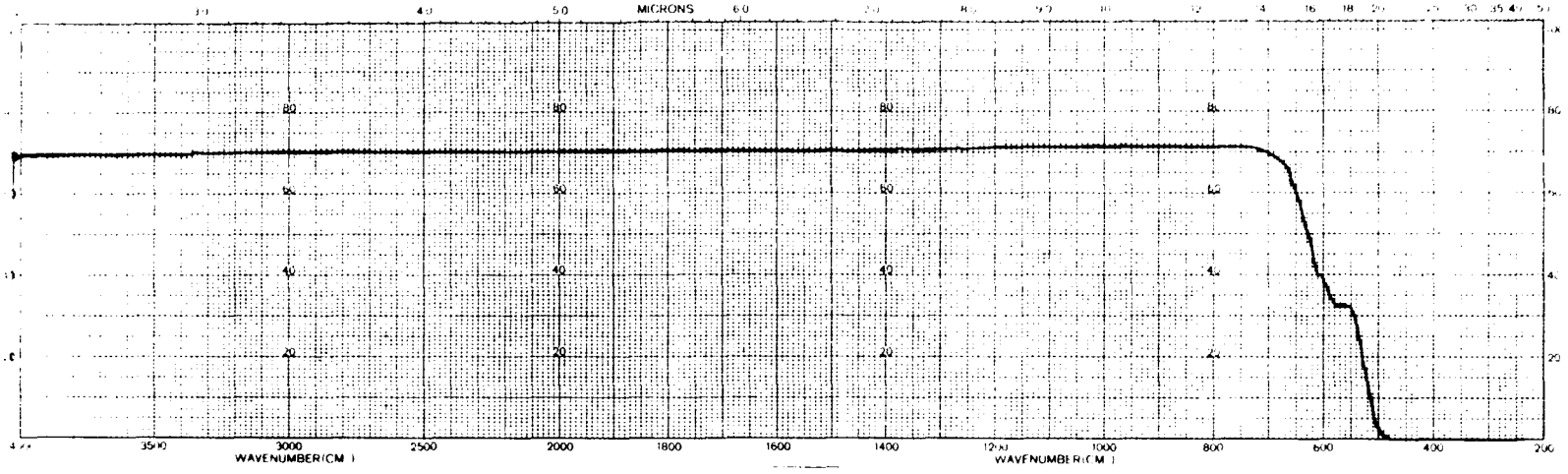

Figure 33. Infrared Transmittance, Sample No. 7, Pilot Run.

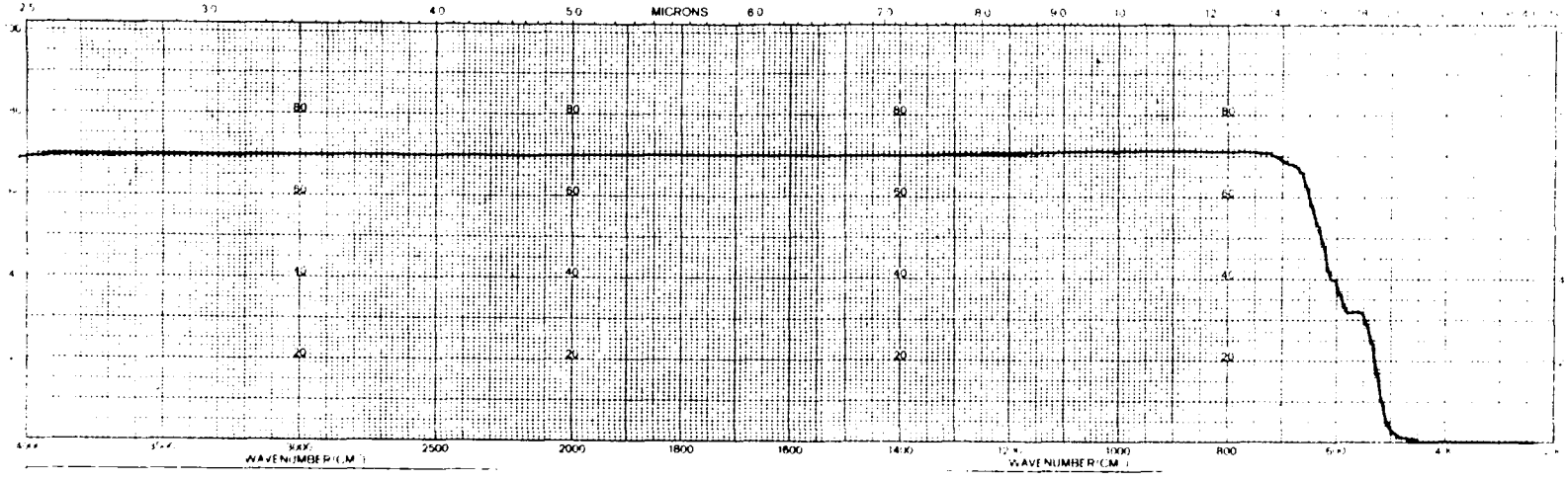

Figure 34. Infrared Transmittance, Sample No. 8, Pilot Run.

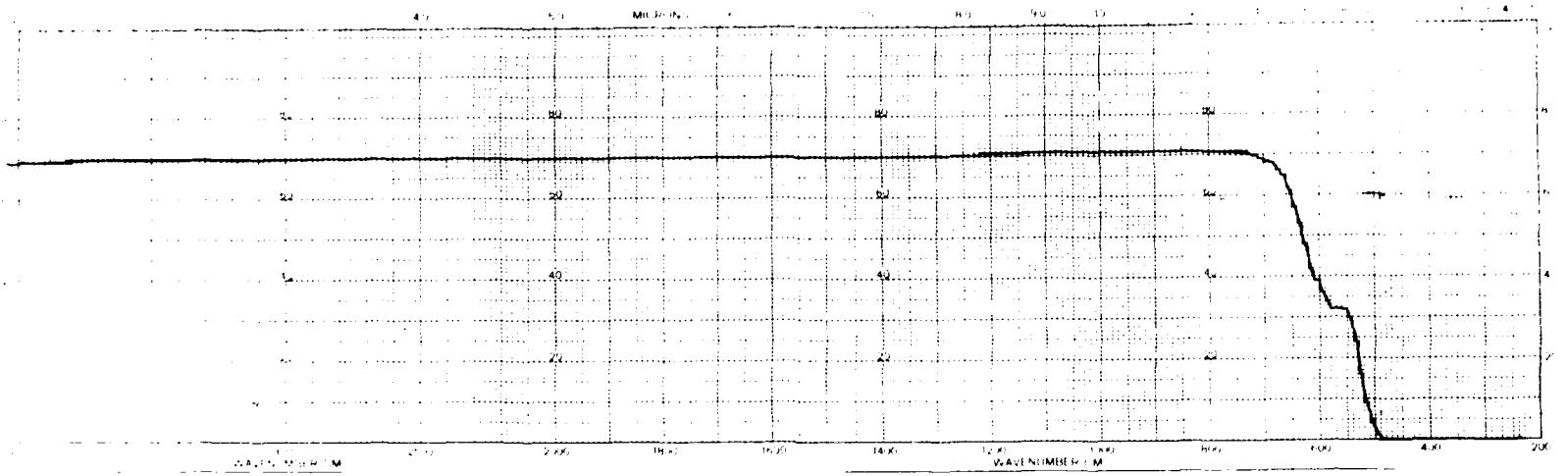

Figure 35. Infrared Transmittance, Sample No. 9, Pilot Run.

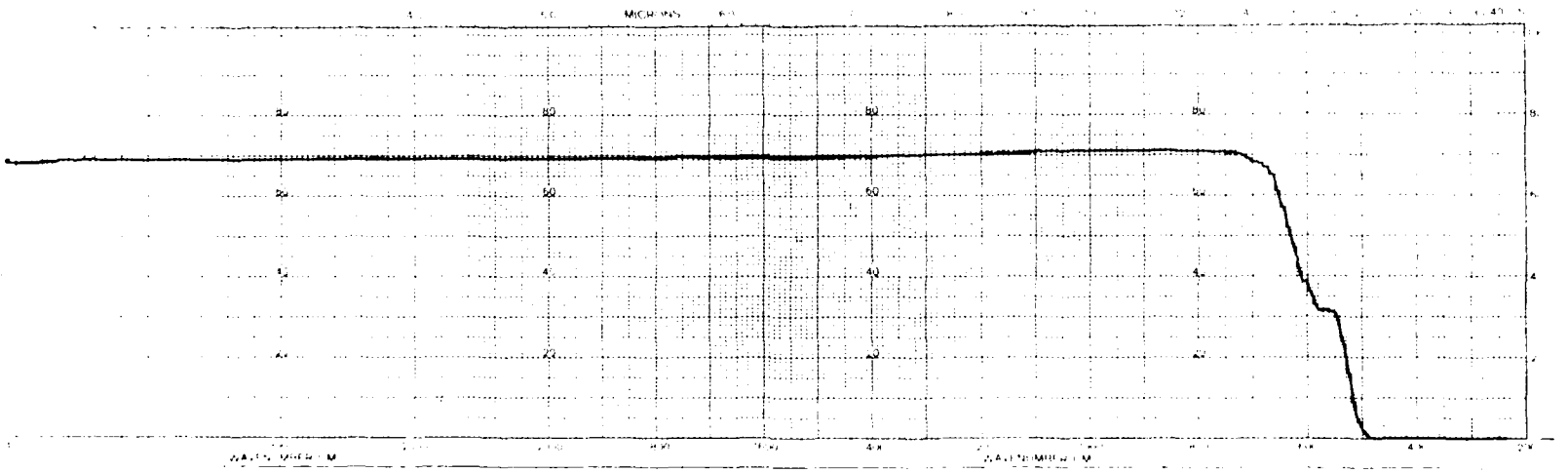

Figure 36. Infrared Transmittance, Sample No. 10, Pilot Run.

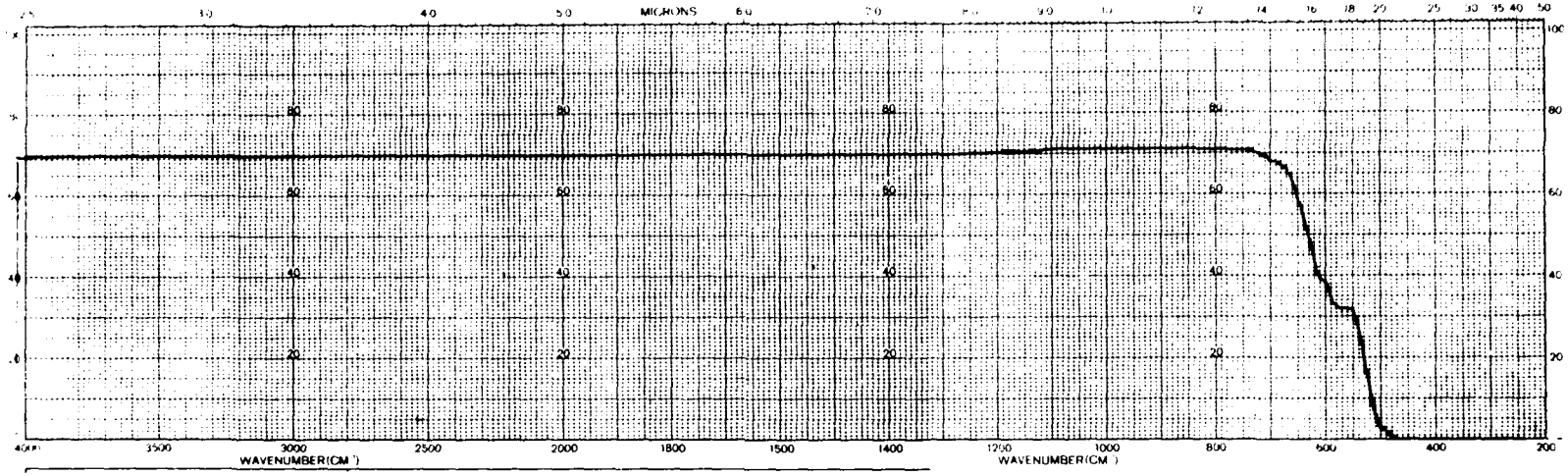

Figure 37. Infrared Transmittance, Sample No. 11, Pilot Run.

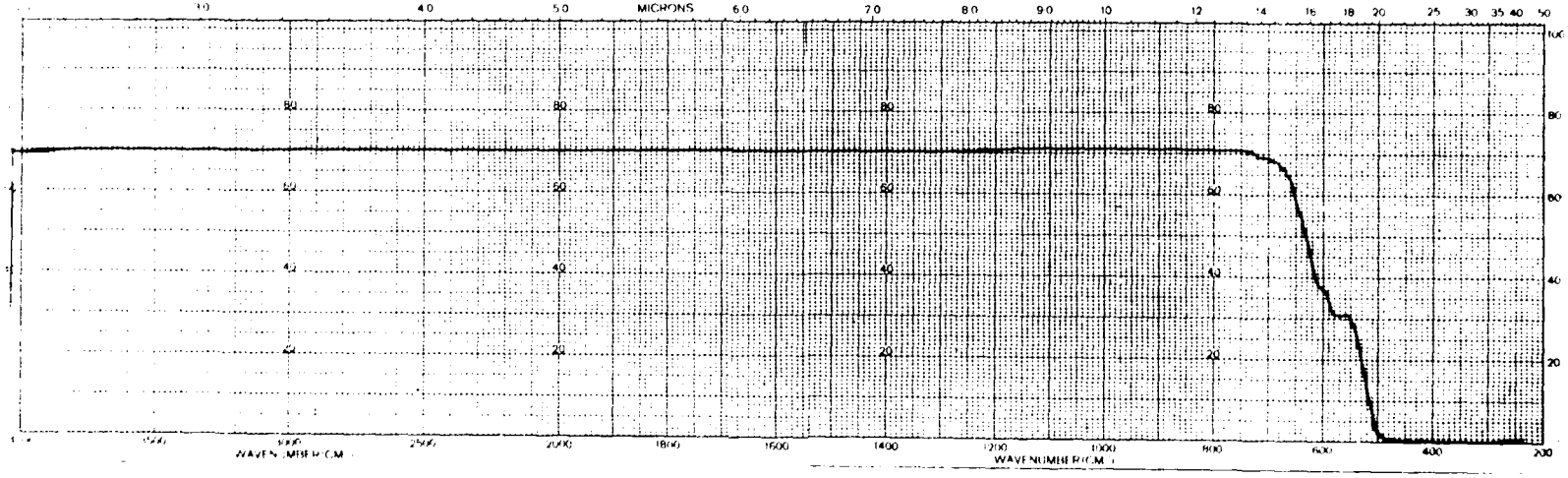

Figure 38. Infrared Transmittance, Sample No. 12, Pilot Run.

TABLE 8

ABSORPTION COEFFICIENT AT 10.6 μm*

FOR PILOT RUN

Specimen No.	Absorption Coefficient (cm^{-1})
1	0.0016
2	0.0019
3	0.0012
4	0.0024
5	0.0017
6	0.0018
7	0.0018
8	0.0018
9	0.0018
10	0.0022
11	0.0019
12	0.0015

*Includes surface absorption

Flexural test specimens, approximately 1/4 by 2 inches, were fabricated from top and bottom areas of the deposit and tested for modulus of rupture. Table 10 presents the data, showing adherence to the specification (minimum strength of 6570 and average strength of 7300 psi).

The twelve polished samples from the Pilot Run were tested and found to meet dimensional requirements, parallelism, chip and fracture criteria.

Surface hardness measured on witness samples was 105 Knoop, using a 50 kg load, meeting the specification.

No relative retardation was observed in any of the twelve samples when tested for strain.

TABLE 9

IMAGE SPOILING DATA FOR PILOT RUN

Sample No.	Image Width @ 50% Intensity (μrad) No Sample	With Sample	% Image Growth
	8 to 12 μm		
1	202.3	203.1	0.4
2	198.6	201.3	1.4
3	202.7	203.8	0.5
4	200.1	202.7	1.3
5	201.2	200.9	---
6	203.4	206.6	1.6
7	198.1	198.9	0.4
8	200.6	203.1	1.2
	0.6328 μm		
1	14.8	15.2	2.7
2	14.8	14.7	---
3	13.9	13.7	---
4	14.0	15.7	12.1
5	14.6	15.5	6.2
6	15.1	16.4	8.6
7	15.5	15.9	2.6
8	14.2	14.7	3.5

TABLE 10

FLEXURAL STRENGTH OF PILOT RUN

(4-point loading)

TOP

Sample No.	Strength (X 10^3 psi)
1	8.2
2	7.1
3	9.6
4	8.8
5	9.5

BOTTOM

1	7.3
2	7.1
3	8.5
4	7.3
5	8.1

Avg. 8.2 ± 0.9

3.0 CONCLUSIONS

For the past eighteen months, Raytheon Company has been involved in a manufacturing and methods technology program for the establishment of a fully automated production process for the fabrication of high quality zinc selenide optical blanks for windows and lens elements. The purpose of the program was to further develop techniques to mass produce large quantities of lens blanks at a reduced cost.

The program was divided into three phases. In the first phase of the program using a Raytheon designed external zinc source, whose feasibility had been demonstrated on experimental deposits, zinc selenide was deposited and evaluated. In Phase II of the program lens blanks were produced by the fully automated process and the resulting material was evaluated to determine if it met the specifications set forth by the Night Vision and Electro-Optics Laboratory. The third phase of the program demonstrated the production capability of a pilot line to manufacture high quality zinc selenide blanks at a rate of four hundred and eighty-one (481) units per month.

The results of the program indicate the fully automated process yields material whose optical and mechanical properties exceed the requirement of the program. Further, it was demonstrated that eight hundred lens blanks could be manufactured per month. Overall yield efficiencies are projected to be at least sixty (60) percent. Thus a minimum of four hundred and eighty (480) lens blanks can be fabricated per month. Further, because of the increased size of the deposition mandrels, the "design to shape" concept, and improved efficiencies on a production basis, the cost of a lens blank can be reduced in production size lots by approximately sixty (60) percent.

APPENDIX A

PROCESS SPECIFICATION

ZINC SELENIDE LENS BLANKS

PROCESS SPECIFICATION
ZINC SELENIDE LENS BLANKS

1.0 SCOPE

This process specification describes the procedure used for the production of CVD zinc selenide lens blanks at the Raytheon Resarch Division under Contract No. DAAB07-78-C-2038.

2.0 APPLICABLE DOCUMENTS

None.

3.0 REQUIREMENTS

3.1 Deposition System Setup

3.1.1 Equipment

3.1.1.1 Raytheon designed 51 inch dia. vacuum furnace and control systems.

3.1.1.2 Raytheon designed zinc feeder assembly & evaporator.

3.1.1.3 Industrial vacuum system.

3.1.1.4 Laboratory scale with 200 lb capacity.

3.1.1.5 H_2Se nozzle assemblies.

3.1.1.6 Mandrel assembly.

3.1.1.7 Exhaust assembly.

3.1.1.8 Hydraulic lift 6000 lb capacity.

3.1.1.9 Two 3/4 inch ratchet wrenches.

3.1.1.10 Four graphite felt insulation pads, 4-in. thick, 48 in. dia.

3.1.1.11 1/2 ton chain hoist.

3.1.1.12 Pressure sensing console - 0.1 mm detection - Serta Systems Model 204 capacitance transducer and Raytheon digital readout system.

3.1.1.13 House argon supply - 500 gal liquid capacity.

3.1.1.14 Raytheon exhaust gas scrubbing tower system - min. capacity 12 lpm H_2Se.

3.1.1.15 "Snoop" leak detecting fluid - 8 fl. oz. applicator bottles.

3.1.1.16 G.E. Type H-2 halogen leak detector.

3.1.2 Materials

3.1.2.1 Zinc wire - 99.99 percent pure, grease free.

3.1.2.2 Mold releasing agent, bulk 30 lb containers.

3.1.2.3 H_2Se gas, 1-A cylinders, 99.99% pure.

3.1.2.4 Argon liquid, 99.998% pure.

3.1.2.5 KOH, 55 gal drums commercial grade, 45% solutions.

3.1.2.6 10% $CuSO_4$ solution.

3.1.3 Safety Precautions

3.1.3.1 Hard hats must be worn while working in the furnace area during setup of the furnace.

3.1.3.2 Toxic gas tanks (H_2Se) are stored and used in toxic gas equipped storage shed external to the furnace room.

3.1.3.3 Unspent toxic gases (H_2Se) are removed from the furnace exhaust in the KOH exhaust gas scrubbing system.

3.1.3.4 Goggles, gloves and protective outer garments are worn during KOH handling operations.

3.1.4 Procedure

3.1.4.1 Vacuum clean zinc container.

3.1.4.2 Load designated weight of zinc wire in container.

3.1.4.3 Affix container covers and gaskets and tighten cover bolts.

3.1.4.4 Install H_2Se nozzles.

3.1.4.5 Apply graphite releasing agent to mandrel assembly parts.

3.1.4.6 Assemble mandrel in place.

3.1.4.7 Install exhaust assembly.

3.1.4.8 Install evaporation assembly.

3.1.4.9 Raise bottom flange with evaporator and mandrel assemblies into furnace.

3.1.4.10 Secure flange with sixteen 1/2-16 bolts.

3.1.4.11 Install top graphite felt insulation from top platform.

3.1.4.12 Lower top flange into place.

3.1.4.13 Connect vacuum line to furnace exhaust port.

3.1.4.14 Install top and two side T/C.

3.1.4.15 Connect H_2Se gas lines from flow panel to H_2Se nozzle assemblies.

3.1.4.16 Connect zinc feeder mechanism to proper port connecting to evaporator.

3.1.4.17 Install wire from container to feeder mechanism.

3.1.4.18 Install five evaporator T/C.

3.1.4.19 Connect evaporator heater power cables to proper heater electrode feedthrough ports.

3.1.4.20 Connect water coolant lines to top and bottom flanges and power ports.

3.1.4.21 Weigh H_2Se tanks (no. of tanks designated on work ticket) and record weight and data on tank.

3.1.4.22 Connect proper amount of tanks to H_2Se gas manifold located in the "toxic gas" room.

3.1.4.23 Check liquid argon level (house argon gas supply source). If level is below "5/8" level have bottle filled.

3.1.4.24 Fill two KOH solution reservoirs in exhaust gas scrubbing system. Each tank 27-½ gal, 45% KOH solution and 90 gal water.

3.1.4.25 Fill two H_2Se indicator bulbs with 1/2 gal, 10% $CuSO_4$ solution.

3.1.4.26 Bakeout loaded furnace. Main furnace power only - manually control - heat to 100°C and hold for 1 hr. Turn on furnace vacuum pump with vacuum line isolated from furnace.

3.1.4.27 After 1 hr bakeout, shut off furnace power, open furnace (over 1 hr period of time) to full vacuum and pump on furnace for 2 hrs.

3.1.4.28 Isolate furnace and record rate of furnace pressure rise over 1 hr period.

3.1.4.29 Backfill entire furnace, gas lines, flow meters with argon to a positive pressure $\sim$ 800 mm. Leak check entire furnace system for large leaks using "Snoop" leak detecting fluid. Correct each leak as it is located.

3.1.4.30 Pressurize furnace again to $\sim$ 800 mm with argon and 5% halogen gas. Leak check for minor leaks with halogen leak detector. Correct each leak as it is located.

3.1.4.31 Pump down the furnace to full vacuum >>1.0 mm and hold at full vacuum for 2 hrs. Record vacuum readings on main furnace exhaust line and nozzles.

3.1.4.32 Isolate furnace from pump and record rate of pressure rise over 1 hr period of time. If pressure rise is greater than 0.2 mm/hr repeat furnace leak check steps.

3.1.4.33 Turn on KOH exhaust scrubbing system.

3.1.4.34 Isolate H_2Se gas line from H_2Se tank manifold from the flow panel.

3.1.4.35 Open all H_2Se gas tanks on the manifold and increase all of the tank gas regulators to a line pressure of 30 lbs.

3.2 Running Deposition

3.2.1 Equipment

3.2.1.1 Log sheets A and B.

3.2.1.2 Spare exhaust line filter cartridge - Dollinger Model GP-123-32S, 1020 CFM capacity.

3.2.1.3 Auxilliary vacuum pump, Welch Scientific Model No. 1402, 5 CFM capacity.

3.2.2 Materials

No additional material requirements during the deposition period.

3.2.3 Safety Precautions

3.2.3.1 Scott emergency air packs are available at all entrances to furnace room with 20 to 30 minute air supply.

3.2.3.2 Furnace room is equipped with an emergency exhaust fan with capacity of 15,000 CFM.

3.2.3.3 Residual H_2Se indicator solutions are provided at final furnace exhaust. These are continuously monitored by furnace operations.

3.2.3.4 Two men on duty during entire deposition period.

3.2.4 Procedure

3.2.4.1 Start argon to H_2Se nozzle, and argon to evaporator at 1.0 lpm.

3.2.4.2 Throttle vacuum pump to control furnace pressures at 600 mm Hg.

3.2.4.3 Start main furnace power on "manual" and bring mandrel temperature to control temperature designated on work ticket. Heatup takes 12-20 hrs. Control furnace at temperature by switching to "auto control."

3.2.4.4 Start evaporator heater and bring evaporator to temperature designated on work ticket. Control at temperature on "auto control."

3.2.4.5 Start multipoint recorder (12 position). Continuously record two evaporator pressures, two evaporator temperatures, two furnace temperatures, and one line pressure.

3.2.4.6 Reduce furnace pressure to operating pressure (designated on work ticket usually mm Hg). Switch to auto pressure control at this pressure.

3.2.4.7 Increase argon flows to "operating flows" as designated on work ticket.

3.2.4.8 Turn on wire feed mechanism at rate prescribed on work ticket. Monitor evaporator pressure and temperature until steady-state is maintained. Process takes 1 to 3 hrs.

3.2.4.9 Set H_2Se controllers to flow values designated on work ticket, then open H_2Se isolation valve to flow panel and adjust line H_2Se regulator to 6 psi.

3.2.4.10 Record deposition hr "0" on log sheets A and B, and record all flows, pressures, temperatures, and power as designated. Repeat check all readings every 1/2 hr during depositon period.

3.2.4.11 When pressure differential of 2 mm Hg develops across exhaust line filter cut in parallel filter, then isolate dirty filter and change filter cartridge. Filter changes occur every 100 hrs under normal conditions.

3.2.4.12 Evacuate original filter with auxilliary pump and leave ready for next filter change. Vacuum clean dirty filter cartridge with house vacuum system and leave ready for next filter change.

3.2.4.13 At the end of deposition period (120-160 hrs) designated on work ticket, shut off H_2Se gas at each tank valve.

3.2.4.14 Reduce argon flows to 1.0 lpm.

3.2.4.15 Increase furnace pressure to 600 mm Hg.

3.2.4.16 Shut off evaporator power supply.

3.2.4.17 Slow cool mandrel at rate of 10°C/10 min. to 200°C, then shut off main furnace power and let mandrel cool with furnace.

3.2.4.18 When furnace is @ 200°C shut off all gas flows and pump furnace at full vacuum for minimum of 12 hrs.

3.2.4.19 Shut off the exhaust gas scrubbing system.

3.3 Disassembly

3.3.1 Equipment

3.3.1.1 1/2 ton chain hoist.

3.3.1.2 10 to 20 cu ft paper batting.

3.3.1.3 Hydraulic lift, 6000 lbs capacity.

3.3.1.4 Hydraulic lift truck, 1000 lb capacity, Lee Engineering Co. Model M-252-1.

3.3.1.5 Eight plate removal racks, Raytheon Company.

3.3.1.6 Lyons storage shelving, 3 x 4 ft shelves.

3.3.1.7 Laboratory floor scale - 350 lb capacity.

3.3.1.8 Six empty 55 gal capacity drums, poly liner.

3.3.2 Materials

No additional materials are required during the disassembly process.

3.3.3 Safety Precautions

3.3.3.1 Hard hats must be worn in the furnace area during disassembly operation.

3.3.3.2 Guard rails must be in place when bottom flange hydraulic lift is in operation.

3.3.3.3 Safety goggles, rubber gloves and protective outer garments must be worn while working with KOH in the scrubbing system area.

3.3.4 Procedure

3.3.4.1 Pressurize furnace to atmospheric pressure.

3.3.4.2 Remove three T/C from top and sides of the furnace.

3.3.4.3 Remove exhaust line from top flange of furnace. Vacuum clean exhaust line using house vacuum system.

3.3.4.4 Remove water cooling line to top and bottom flanges.

3.3.4.5 Lift off and remove top access flange using 1/2 ton chain hoist.

3.3.4.6 Vacuum clean loose excess zinc using house vacuum system.

3.3.4.7 Remove top graphite felt insulation.

3.3.4.8 Remove exhaust assembly.

3.3.4.9 Pack area between deposited plates with paper batting to secure for removal from furnace.

3.3.4.10 Remove all input gas lines from bottom flange assembly.

3.3.4.11 Secure bottom flange with 6000 lb capacity hydraulic lift, then remove 16 fastening bolts.

3.3.4.12 Lower bottom access flange with retort and mandrel assemblies.

3.3.4.13 Position portable hydraulic lift at mandrel plate A. Position plate removal rack on the lift truck.

3.3.4.14 Lower mandrel plate A with CVD zinc selenide plates onto rack. Lower rack and plate using hydraulic lift.

3.3.4.15 Repeat 3.3.4.13 and 3.3.4.14 for (3) additional mandrel plates.

3.3.4.16 Vacuum clean, using house vacuum, evaporator section and bottom flange.

3.3.4.17 Disconnect H_2Se tanks from H_2S manifold system in the toxic gas shed. Weigh each tank and record weight on tank and weight loss during the run on log sheet A.

3.3.4.18 Drain spent KOH from KOH reservoir into 55 55 gal drums. Mark drums "Used KOH ZnSe."

3.4 Evaluation Testing

3.4.1 Equipment

3.4.1.1 Ultrasonic gauge, Panametrics, Inc., Model 5222.

3.4.1.2 Diamond cutoff saw, Felker Di-Met, Model 120-B, 12" blade.

3.4.2 Materials

No additional materials are required for evaluation processing.

3.4.3 Safety Precautions

3.4.3.1 Adequate ventilation is provided in the cutting and grinding area.

3.4.3.2 Face shield and outer garments are worn during cutting operation.

3.4.4 Procedure

3.4.4.1 The plate thickness is measured and recorded using ultrasonic thickness gauge.

3.4.4.2 Optical test specimens ($\sim$ 1 X 1 X maximum in.) are taken from the top, middle, and bottom of deposited plate.

3.4.4.3 Infrared transmittance and absorption coefficient are measured for each sample.

3.4.4.4 Select and cut \3 X 6 in. area from top and bottom of a plate for processing into tensile test bars.

3.4.4.5 Beams are tested for flexural strength in 4 point loading over a 3 in. span.

3.4 4.6 Select and cut any additional test samples required.

3.4.4.7 Cut or core lens blank as required.

LOG SHEET

PAGE NO. _____ "A" RUN NO. _____ FURNACE _____ W.O. NO. W_____ DATE_____

Time of Reading	Dep. Hrs	FURNACE					EVAPORATOR					Remarks
		Temperature		Press. mm Hg	Power		Temperature		Press. mm Hg	Power		
		M.V.	°C		Volts	Amps	M.V.	°C		Volts	Amps	

LOG SHEET

PAGE NO._____ "B" RUN NO._____ FURNACE _____ W.O. No. W-_____ DATE_____

Time of Reading	Dep. Hrs	GAS FLOWS (SCALE)												REMARKS
		ARGON							H_2Se					
		Inlet	Inlet	Inlet	Inlet	Inlet	Evap	Evap	Inlet	Inlet	Inlet	Inlet	Inlet	

APPENDIX B

QUALITY CONTROL STATIONS

QUALITY CONTROL STATIONS

1. MANDREL PREPARATION

Mandrel surfaces exposed to the deposition are checked for flaws. No large scratches or machining marks on surfaces are permitted. If scratches are present, mandrel surfaces are sanded until smooth.

A graphite mold release is applied to the deposition of the mandrels. A minimum of 0.001 in. of mold release is required.

Mandrels are not assembled until above inspection is passed. - 100% yield

2. ASSEMBLY OF DEPOSITION SYSTEM

The entire deposition system is assembled upon the bottom flange of the furnace. Prior to loading the system into the furnace, an inspection is conducted to insure all subsystems are included and aligned properly in the system. Thermocouples and heaters are checked for proper electrical resistivity. Gas-carrying components are checked for restrictions to flow and connections are checked for leaks under normal flow conditions.

The next phase of loading the system into the furnace is not conducted until a 100 percent inspection of all components is conducted. - 100% yield

3. LEAK CHECKING

The leak checking procedure is carried out as described in Sections 3.1.4.27 to 3.1.4.32 of the Process Specification. Deposition stage of the zinc selenide blanks does not begin until leaks are repaired and the pressure rise in the furnace is 0.2 mm of Hg/hr. - 100% yield

4. DISASSEMBLY OF FURNACE

Disassembly of the furnace is conducted according to Procedure 3.3 of the Process Specification. Anticipated yield due to cracking upon cooling of deposited material and handling of mandrel assembly is - 75%.

5. EVALUATION AND CUTTING

Optical and mechanical test specimens are located and cut from top, middle and bottom sections of the deposit. Measurements are conducted to ensure material meets property requirements as per SCS-524 of Work Statement.

Upon verification of property compliance, individual lens sites are cored or cut, isolating them from the deposited plates. - 80% yield.

APPENDIX C

TEST PROCEDURES AND FACILITIES

TEST PROCEDURES AND FACILITIES

A. Inclusions

Each sample shall be tested in accordance with the Method 1 test procedure for inclusions of MIL-G-174.

B. Surface Hardness

The hardness test shall be conducted using a Riehle Kentron microhardness tester, Model No. AK 208 or equivalent apparatus. The average Knoop microhardness will be measured using a 50-gram load. Witness samples shall be used for this measurement.

C. Transmittance

Transmittance shall be measured by comparing the amount of energy falling on a detector, having focusing optics from a beam with and without a sample in the path of the beam. A chopper may interrupt the beam so that synchronous detection and amplification may be made. This measurement will be made on a Carey Model 14 spectrophotometer for the 0.6 to 1.1 um range and a Perkin Elmer Model 457 spectrophotometer for 2.5 to 40 um range. If more precise measurements are necessary, a Perkin Elmer Model 180 will be used. Witness samples may be used for this measurement.

D. Absorption

The absorption of Raytheon CVD ZnSe at 10.6 um is too small to be measured by the method of MIL-G-174. We propose instead that the absorption be measured on witness samples by the now well-established calorimetric technique. Our calorimetric test apparatus includes a CO_2 laser capable of up to 50 W output, a vacuum chamber in which the test sample is mounted,

and a power meter which measures the power transmitted through the sample. Figure 1 displays a schematic drawing of the calorimeter.

Figure 2 depicts the power flow in a test sample. Power enters the sample at the left-hand surface and an amount P_T exists at the right-hand

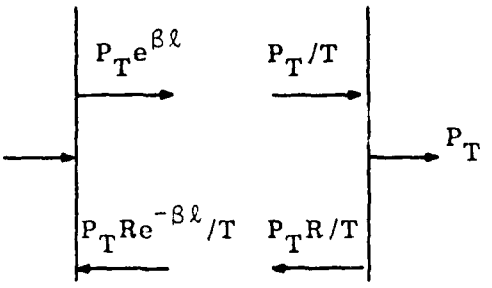

Figure 2

surface. It is well known that when electromagnetic radiation is incident near normal on a surface between a dielectric and air a fraction R is reflected, given by

$$R = \left(\frac{N - 1}{N + 1}\right)^2 \quad ,$$

where N is the index of refraction of the dielectric. The fraction transmitted, T, is given by 1 - R. Applying these ideas to the right-hand surface, we conclude that the power incident on that surface is P_T/T, and the power reflected from it is $P_T R/T$.

The power abosrbing property of a bulk material is usually characterized by an absorption coefficient, β, defined by the statement that the power traversing a distance X in the material is reduced by the factor exp $(-\beta X)$. Thus, the power arriving at the right-hand surface originates at the left-hand surface at a level P_T exp $(\beta \ell)/T$, and the power reflected at the right-hand surface arrives at the left-hand surface with the level $P_T R$ exp $(-\beta \ell)/T$, where ℓ is the thickness of the sample. Then, the power absorbed in the sample is

$$P_{abs} = \frac{P_T}{T} \left[\exp(\beta \ell) - 1 + R\{1 - \exp(-\beta \ell)\}\right] \quad .$$

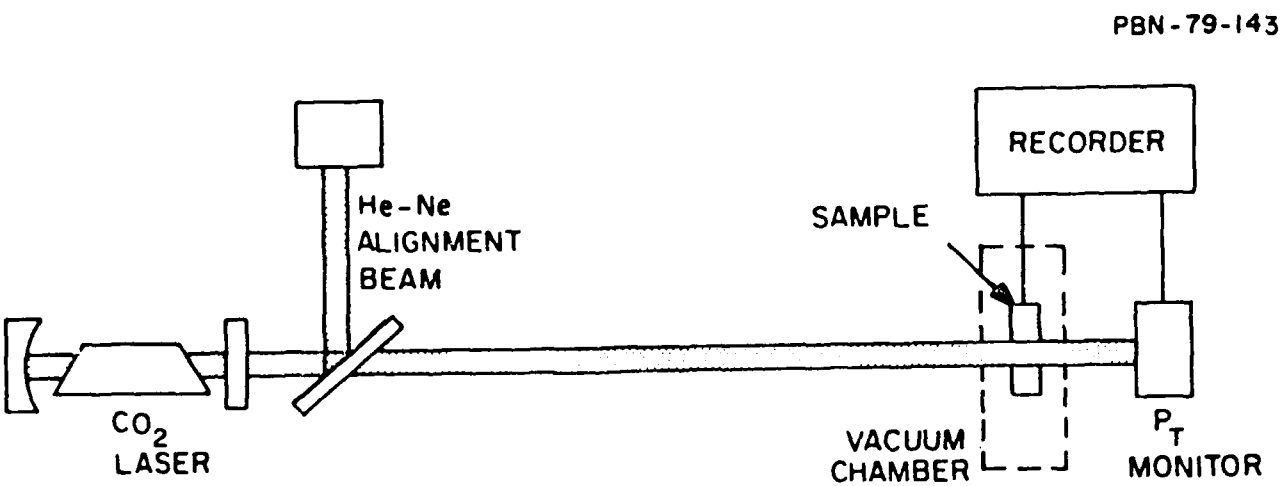

Figure 1. SCHEMATIC DRAWING OF 10.6 μm CALORIMETER

This expression can be solved for $\beta\ell$ so that, knowing the transmitted power, the absorbed power, the refractive index, and the thickness, the absorption coefficient can be calculated. The exact expression is somewhat complicated and will not be given here, although it is, in fact, used in our measurements. For $\beta\ell << 1$, the following expression may be obtained:

$$\beta\ell = \frac{2N}{N^2 + 1} \frac{P_{abs}}{P_T} \quad .$$

The power transmitted (P_T) may be measured directly with a laser power meter. The most exacting part of laser calorimetry is the measurement of the power absorbed by the sample (P_{abs}). In our measurements P_{abs} is determined from the rate of change in temperature of the sample. A thermocouple is held in contact with the sample surface and, with suitable amplification, monitors the temperature of the surface. If the rise in sample temperture per unit time is dT/dt, the power absorbed is given by

$$P_{abs} = Jmc \frac{dT}{dt} \quad ,$$

where m is the sample mass, c the specific heat of the sample, and J the mechanical equivalent of heat.

This derivation assumes that no heat transfer occurs between the sample and its surroundings. Although measures are taken to minimize this transfer, it is of course not zero. However, corrections can be obtained from the rate of temperature change observed in the initial drift phase (before the laser beam is turned on) and in the decay phase (after the laser beam is turned off).

Representative curves from our calorimeter are shown in Figure 3. The curve marked S is the amplified output voltage of the thermocouple indicating the sample temperature, and the curve marked P is the output of the laser power meter measuring the transmitted power. (The two curves are displaced by half a centimeter along the time axis.) The slope of the sample curve is

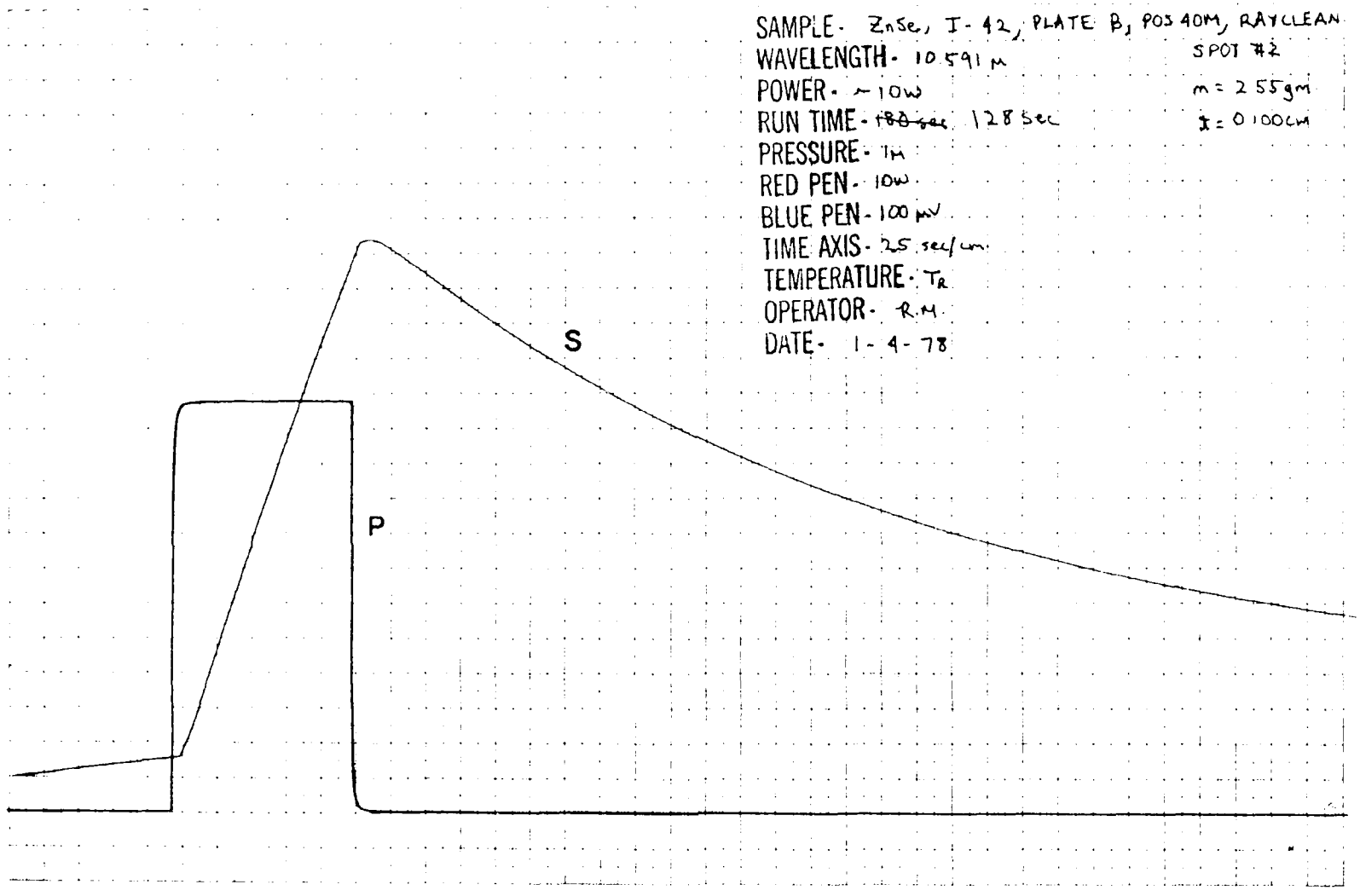

Figure 3. SAMPLE TEMPERATURE VS TIME CURVES FOR CALORIMETRY MEASUREMENTS

proportional to the time rate of change of the sample temperature, dT/dt. Suppose the temperature and rate of temperature change are measured at three points in the drift, heating (laser on) and decay regions. Let these temperatures be T_1, T_2 and T_3 with the corresponding rates of change T_1', T_2' and T_3'. Then, if one assumes that the rate of heat transfer is proportional to the difference in temperature between the sample and its surroundings, one can obtain a rate of temperature change in the heating range corrected for heat transfer given by

$$T_2'_{corr} = T_2' - T_1' \frac{T_3 - T_2}{T_3 - T_1} - T_3' \frac{T_2 - T_1}{T_3 - T_1} .$$

In our measurements, the slopes of the sample curve in appropriate regions are automatically determined by a microcomputer which also controls the opening and closing of the laser shutter and performed the requisite calculations. When the run is begun the record is taken for two minutes before the shutter is opened. It will be seen that for the run illustrated the initial sample temperature is below ambient so that the temperature drifts upward. The shutter is then opened and the sample begins to heat up. After an appropriate time the shutter is closed and the decay phase is monitored for 10 minutes. After the necessary sample parameters are provided, the microcomputer prints out the values of β and the fraction of power absorbed.

E. Scatter (Image Spoiling)

The effect of scattering in the 8 to 12 μm range shall be determined through what we usually refer to as an image-spoiling measurement. Our apparatus, shown in Fig. 4, utilizes the radiation from a blackbody source operating at 875 K. A blocking filter is used to cut off the radiation below 8 μm. The radiation is passed through an adjustable slit typically set at 30 μm. The slit serves as an object for an off-axis paraboloidal mirror of 8-inch diameter and 64-inch focal length, which renders the radiation parallel. A second off-axis paraboloid is used to refocus the collimated beam to an image

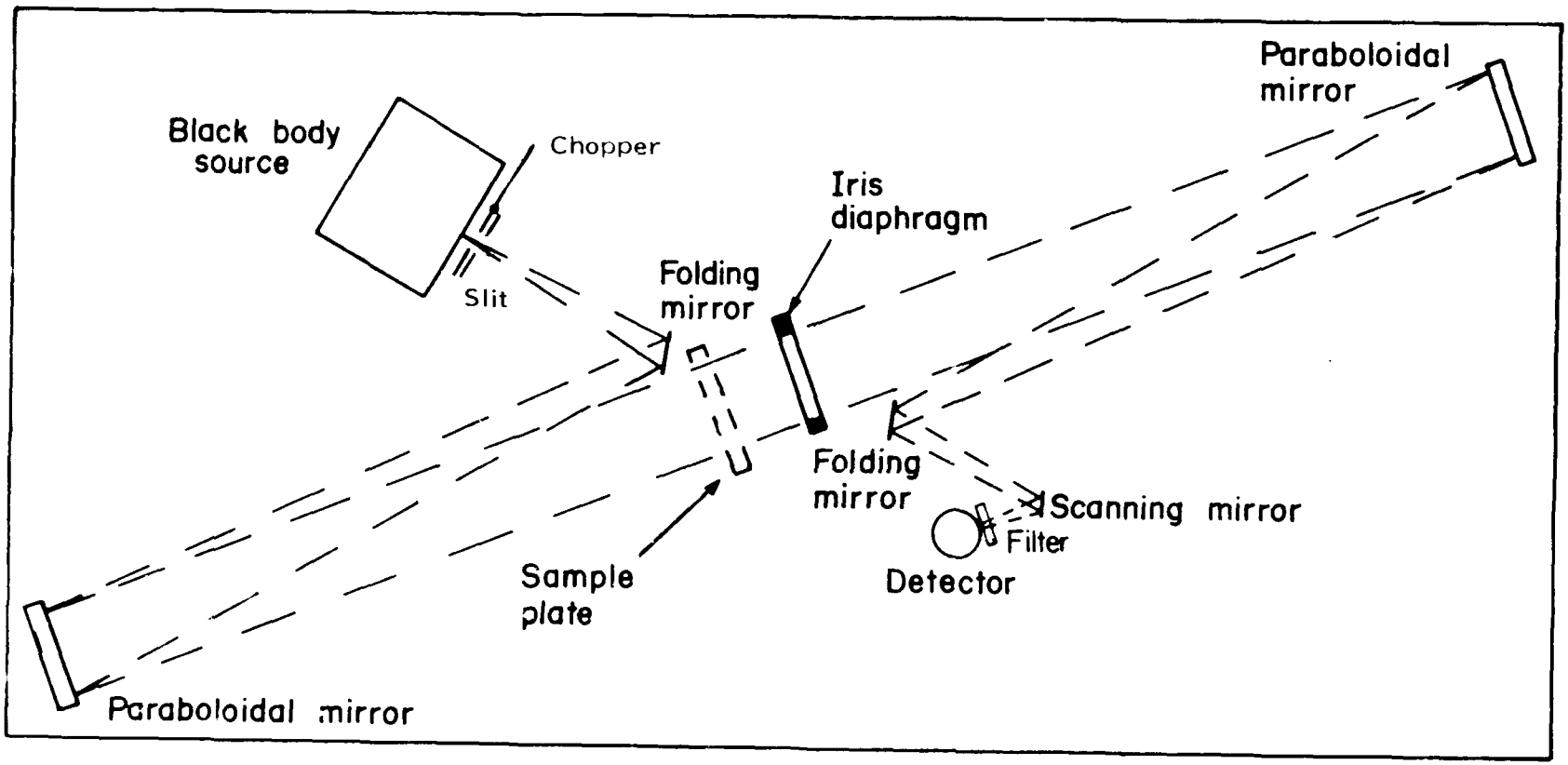

Figure 4. IMAGE SPOILING - MTF APPARATUS

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MANUFACTURING METHODS & TECHNOLOGY PROGRAM, ZINC SELENIDE BLANK--ETC(U)
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of the slit. By means of a scanning mirror system, the image is swept across the detector at a rate of approximately 60 Hz. The detector uses a mercury cadmium telluride element 0.001 inch wide and 0.020 inch high, the spectral response of which cuts off above 12 um. The amplified detector output is applied to a signal averager, which builds up an acceptably noise-free redord of the intensity distribution across the image in approximately a minute.

By examining this distribution, a suitable measure of the image width may be obtained. This is illustrated in Figure 5, showing a typical output curve obtained after smoothing in the signal averager. We have commonly worked with the image width at 15 percent and 50 percent of peak, but other values (e.g., 10 percent) can be used. We can also perform a Fourier transfer on the image curve and obtain the modulation transfer function (MTF) for the system. To test the image-spoiling property of a polished plate, measurements are made with and without the plate placed in the collimated beam between the two paraboloids, and the extent to which the width of the image is increased or the MTF decreased by the presence of the plate is observed.

Comparable measurements are made in the visible region with suitable modifications to the source, slit and detector. For the source we use a He Ne laser operating at 632.8 nm or a broadband quartz halogen lamp. The slit width is of the order of 5 um. Since no detector of sufficiently narrow width exists for the visible measurement, the image is scanned across the edge of a razor blade and the level of power passing the edge at any time is determined with a photomultiplier tube. The curve thus obtained is the integral of the image intensity distribution and is differentiated by computer before being analyzed as in the infrared measurement.

F. Modulus of Rupture

Modulus of rupture test samples shall be fabricated from the confirmatory deposit. A minimum of ten samples will be tested. The samples will be beams of 6 x 6 x 60 mm in size. All surfaces will be polished and the corners will

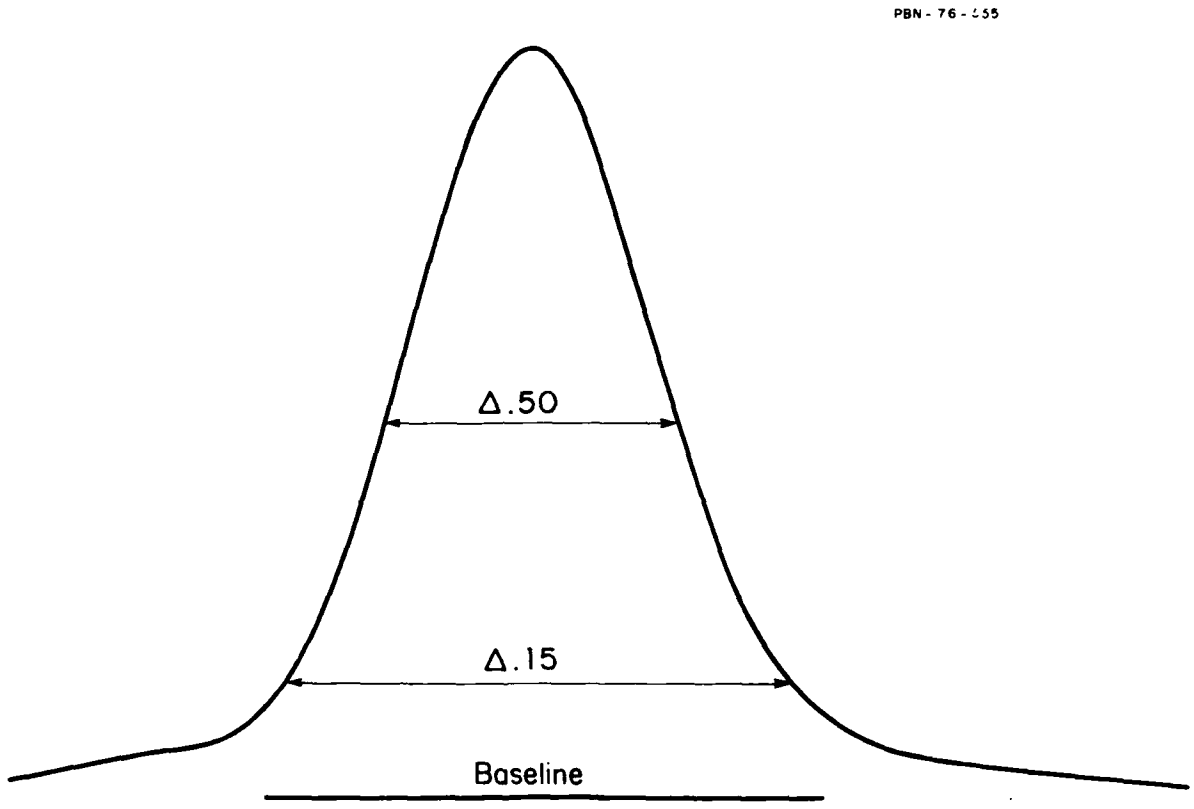

Figure 5. TYPICAL IMAGE SPOILING CURVE

be rounded and polished. The sample location will be within reasonable proximity to the zinc selenide blanks and recorded in the test report.

Modulus of rupture tests will be performed on the test beams using an Instron Model TT-C-M1 testing machine. Four-point loading over a 2-inch span will be used. Figure 6 displays the loading diagram for this test. The modulus of rupture, S_R, will be determined by the equation:

$$S_R = \frac{Mc}{I} = \frac{3P}{2bh^2}$$

Where M = bending moment = $\frac{\text{Load (P)}}{2} \times \frac{\text{Span}}{4}$

I = moment of inertia for the cross section = $\frac{bh^3}{12}$

C = distance of neutral axis to outermost fiber = $\frac{h}{2}$

b = width of sample

h = height of sample

P = breaking load.

G. Parallelism

Each sample will be tested in accordance with the test procedure for "wedge" of MIL-G-1366.

H. Strain

A Polarizing Instrument Company, Model 104 Polarimeter-Polariscope, shall be used to determine the relative retardation of transmitted light through the zinc selenide blanks.

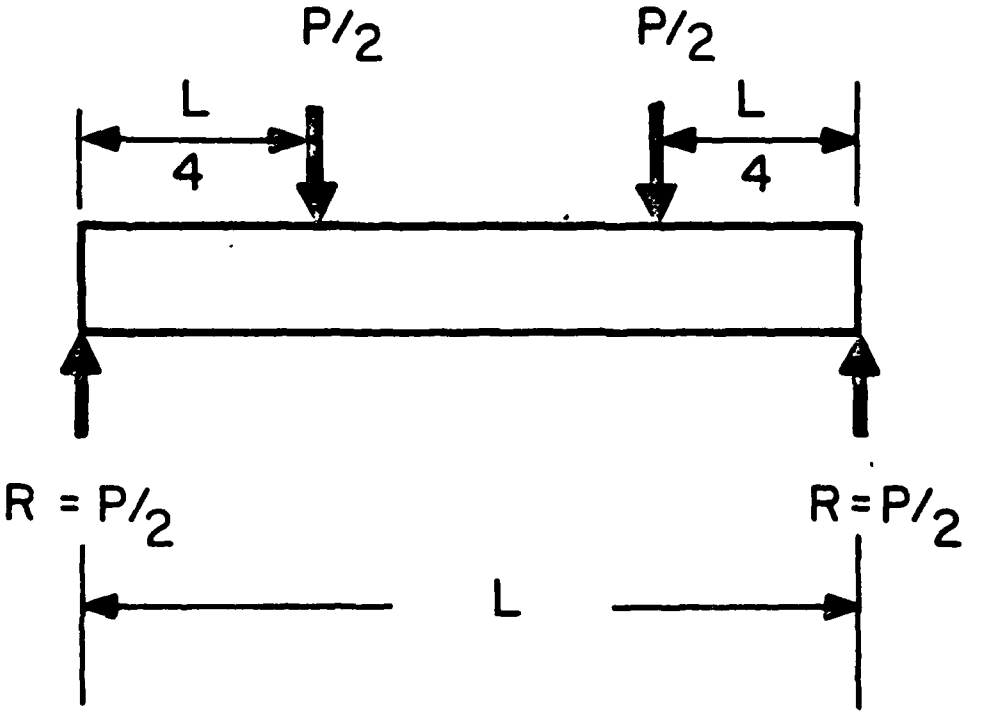

Figure 6 Loading Diagram for Modulus of Rupture Test.

I. Chips and Fractures

A visual examination for chips and fractures shall be made. When detected, they will be measured by standard measuring equipment to determine acceptance or rejection in accordance with the applicable requirements.

J. Visual and Mechanical Examination

The blanks shall be examined to verify that the size and thickness and markings are in accordance with applicable requirements.

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